Notebook Series -- Notebooks by Experimenters Other Than Edison
Battery Experiments -- Experimenters' Books
E. E. Dougherty Notebook, N-20-01-19

This notebook was used by E. E. Dougherty during January-August 1920. The entries pertain to the analysis and preparation of various chemical compounds, particularly for storage batteries. Included are tests of chemical samples from the Edison Storage Battery Co. plant at Silver Lake, New Jersey, along with tests of electrolytes. There are also experiments relating to chemicals used in the manufacture of disc records. Several entries contain comments, including transcriptions of Edison's notes, that indicate a collaboration between Dougherty and Edison on some of this work. The book has been used in both directions. The pages at the front are numbered 1-109; some pages are blank. The pages at the back are inscribed C-Z and A1-Z1; these pages are followed by approximately 25 additional pages without inscriptions.

Old Milroy
for Ni & Co
39,000 lbs Ni & 20 oz Co received at Copper Cliff Sept 1/20

90000# per Incd. Fe by H
Made at Silver Lake
actually 90%
This = 16,000 cells per day

Copperman
J.J. & E.C. Dec. 1919

Jan. 19/20

(1)

Prepare 200 litres
Prism Doubly Crystallized
Nickel Sulphate Solution 1.300 Sp Gr
for Mr Hawley's Exp.

1st Tank Dissolving & Treating
21½" dia. x 26½" deep = litres.
Used 20¾" City Water litres @ 10 °C.
+ 37.239 Kilos. Comm. NiSO, 6 Hg

Started to heat 11 A.M., Finish 1st Time
Temp. 90 °C. x Vol 21½ = l.
After adding the Nickel Sulphate
Temp dropped to 78 °C.
and had 20.5" = l. Vol.
Sp. Gr. 1.147 @ 88 °C. {contained trace free acid, Fe & Cu}
Added 1500. grm. $NiCO_3$ to neutralize the free acid
Before treating Vol. 22½" or litre
Then added a few grams $BaCO_3$ to precipitate the Fe
Allowed to settle over night
Vol. = 20 " = 4.166 cu ft 116.3 litre
Sp. Gr. = 1.188 @ 27 °C

1/20/20

(Contin.)

Sol. was neutral to M-O & Blue Lit. mm
contained also traces of oxygen

Note In treating fresh by BaO, Sol. showed the filtered liquor titrating with BaO_2 & working basic.

The above Sol. stood in Crock #1 until 3/15/20 collected some dust, and Bill did not clean Crock #1 properly, also lost a great deal by allowing to flow from filter into trays and tannum line.

* Starting from this point.
Sol. in Crock #1 15" x 21.5"
vol. =

10 cc 1/5 = 256 - 258 cc Key
= $TiSO_4$ gm per L.
$TiSO_4$ # in #2 Crock

(3)

Contin.

2nd Batch
1 Crock full ~~Fermin~~ 8"
= Litres

25 cc 1/10 = 209 - 20 cc Key
= $TiSO_4$
= $TiSO_4 + 6H_2O$

Jan. 20/20

Nickel-Iron Mud Data

~~Another sample which was used from the~~

1000 grams = 880 cc
cu. ft.

1136 grams per liter = 70.89 # per cu ft.

Added 1000 cc H_2O
+ 120 cc HNO_3 (1.19 Sp Gr)

That element is basic
(Sol. said, also showed Fe in sol.)

Pass thru 30 mesh screen
(about 1.2 sp. gr. that)

Vol :- 1780 C.Cs.

Start to settle 2:05 P.M. Finish 5.30
Total time 3 hrs 15 mins

(1st) Vol. of clear sol. 1,420 ccs
Slurry 360 ccs

oversize that would not pass 30m 58. grams } Wet
= 37 cc

principally sand, grains of wood etc

(2nd) Added water again to total vol. 870 cc
Treated and allowed to settle 2 hrs
Clear liquor 520 cc
Slurry 350 cc

Added H_2O again to 870 cc & treated and allowed to settle 2 hrs.

Contd.

(3rd) Clear liquor 560 cc
Slurry 310 cc
Slurry is the flocculated material

In order to see if mud is hard in slurry, or whether it would pass through a mesh filter press

Liquor in Slurry 210 cc
vol of Slurry Press Cake 100 cc
wt of " " 130 gm Wet

14.8165	14.8165
9.0805	12.1960
5.7360	2.6205

Total :- 4.69

wt of Dry Press Cake 70.62 gr
" " Water in " " 59.38 "

Tank #1 For treating up slurry

Tank #2 For settling.

Tank 3 for same purpose.

(Ratio of Mud to total vol. in Tank #1
880 cc, 2000 cc 1 : x = 2.273)

(Continued on page 10)

a) Jan. 20/20

Data for making 300# per week of a.p. $Ni(OH)_2$ for neutralizing

500 cc $NiSO_4$ Sol. Sp. Gr. 1.25 Room temp.
= grms.

Heat
add NaOH sol.

Note Had to throw this out as Nickel preparation was too high and it would not settle.

2nd Exp.
Take 100 cc $NiSO_4$ Sol.
Sp. Gr. 1.195 @ 27 oc.
+ 500 cc H_2O distilled
Heated to boiling and ppted $Ni(OH)_2$ by means of NaOH in xs
Had to increase sol. over all, in order to settle properly, ∴
Total sol. 900 cc
350 cc xs portion left
550 cc liquor removed
or 61.11%
Washed 20 times & settled in x hrs.
(Continued on page 21)

Jan. 20/20

Nickel-Iron Mud Treatment

Contin from p - 7

Tank #1 To be used for treating up Mud, in presence of H_2O & H_2SO_4, so as to have thro 30 mesh to Settling Tanks, & to dissolve all Values.

To be Cylindrical — lead-lined — and to be furnished with lead-coil for heating, also perforated division paddle, & floating discharge pipe.

1 lb. of Wet Mud + H_2O & H_2SO_4 sufficient to dissolve all Values and form a slurry of about 1.2 Sp. Gr. (Wet)

will take 1.10 # H_2O

and 0.2 # H_2SO_4 { 98 by % H_2SO_4 = 66 Bé

In the same ratio (with Margin)

1 cu ft Wet Mud = 2.273 cu ft Slurry 1.2 Sp Gr.

Contin.

Tank 6' dia x 6' deep and keeping abt. 1½' from top will =

197.26 cu. ft.

= 5600 " " Mud

= 3970 lbs. "

3 Chgs per 10 Hrs

= 11910 lbs.

Tank #2 Cylindrical, lead-lined as #1 — to have stirrers and lead steam-coils & floating discharge pipe

Dimensions :- Same as Tank #1.

~~Tank #1 —~~

~~1780 + 1420 + 1920 + 1~~

~~x = [illegible] cu ft~~

Liquor Figures

	Be	%
Orig in Tank #1	1780	100
1st " from " #2	1420	79.77 %
2nd " " " #3	520	29.21 %
3 " " " #4	560	31.46 %
Water added to 1st " Batch	1100	61.79 %

Contin on Page 18

June 23/20

Data for Capacity of Plant
C.P. double xtallized $NiSO_4$
500 # per day

1st Operation

Dissolve to Sp. Gr. 1.188 @ 27°C
ppt. in a 7e

Proportion 1-
320.2 grm $NiSO_4$ xtal to litre
= 19.98 # per cu ft ~~e~~

~~1 litre 1.18 Sp Gr. @ 27°C~~

	Xtals	Vol	Sp Gr	Temp
Original	320.2	1000 cc	1.18	27
Boil to		320 cc	1.56	105
Cool to 25°C in 2 hrs. with stirring	265	160 cc	1.37	27

Add 57 cc H_2O, & heat
to point where supersaturated
Vol of 1st xtals 240 cc
after adding 57 cc H_2O
and boiling, first begin
Cooling 200 cc
Cool to 28°C with stirring 200 73 cc 1.352 27

Contd.

In order to obtain 500 # double
xtallized $NiSO_4$ per day we must
use

	Xtal	%	M.L.	Sp. Gr.	Vol %
2nd Crystal	200	62.30%	73	1.352	7.3%
1st Crystal	265	82.75	160	1.37	26.50%
Original	321	100	1000 original	1.18	100%

500 # per day of C.P. double xtal
= 803 # " " " Coml. Salt

Sol. of 1.18 = 19.98 # per cu ft.
Therefore to accommodate this
wt in the Later Mixing
tank to hold 40.15 cu ft
75% of its depth.

1st or Dissolving Tank

Jan. 26/20

Ni-Fe Mud

Contin. from page 18

Products obtained :-

	Name	Amt.		Room Sp. Gr.
I)	1st Fraction	1,420	cc	1.717
II)	2nd "	570	"	
III)	3rd "	560	"	
IV)	From Liquor	710	"	
V)	" Cake (Hot)	130	gms	
VI)	Over Liq. (")	58	"	

(Analysis)

I	5000-1/10 =	61.6	cc	KOH = Ni	84.393	gms
II	10	37.4	"	" =	9.387	"
III	10	11.4	"	" =	3.079	"
IV	10	10.4	"	" =	0.657	"
V	10.08 gms =	5.56	"	" =	1.485	"
VI					Traces	
			Total		98.991	g

	Ni, gms per L	%
I	59.437	85.75 %
II	18.040	9.47 %
III	5.500	3.11 %
IV	5.070	97.83 % Recovery
V	7.56 %	0.66 %
		1.57 %
		100.00 %

Jan 22/20

500/ Took Coml. $NiSO_4 + 6H_2O$ from Stor — add City water — boil — add 2 qr. abt. of $FeSO_4$ — Add enough BaO_2 to oxidize Fe. — Then add enough $Ba(OH)_2$ to ppt abt 5% of the Ni and all of the Fe — (as there was no $Ba(OH)_2$, BaO_2 was used)

This treatment was supposed to not only neutralize the free H_2SO_4, but also by means of the $Fe(OH)_6$ and $Ni(OH)_2$ ppted, to remove all of the foreign metals such as As — Cu — Pb — Fe — As — Sb — Se — Te — U etc also carry down dirt etc.

The solution was filtered and electrolyzed

Amt 328 cc & Sp. Gr. 1.3

In 3 hrs, deposit was perfect.

1st, deposit very poor, curled.

Anode turned black, probably due to oxygen left in sol.

Formed Chlorides present (continued on page 21)

Jan 24/20

(Contin from p. 8)

300 # C.P. $Ni(OH)_2$ from Doubly x tallizes. $NiSO_4$

To be used for neutralizing (to be marked free of Sol. salts) electrolyte.

Finishes $Ni(OH)_2$ from filter-press received the amount as follows

1.71 gms (wet) = 29.1 in 1 Kg.

= Ni . 1 - 11.60

= $Ni(OH)_2$ 1 - 18.33 %

x Water 1 - 81.67 by diff.

equiv. to $NiSO_4 + 6H_2O$ 51.95%

Vol of $Ni(OH)_2$ press cake 67 gms.

Vol. 6" dia x 1/8" thick

= 3,535 cu. in.

34.6 gms. Coml. $NiSO_4 + 6aq$ made into Sol.

in Say 36 gm. Coml. Salt.

67 gms. : 3,535 cu in :: 300 # = 136080 gms :: x

x = 7181 cu in or 4.16 cu ft

Press in Chambers 24" x 24" x 1" Cake will take care of it in 1 batch

Capac. of Tank

67 # gm. : 1000 # :: 300 # : x = 4477 # H_2O

= 72 cu ft

or Tank 4' dia x 6' deep. (contin on p. 26)

Jan. 28/20

Ni-Fe Mud Exp.

To see if 1st liq. could be used on next batch, and how much Ni it would take up.

Tried in cold also, to see if we could eliminate steam coil in tank.

Added some 1st liq to a fresh batch of mud, and enough H_2SO_4 to give acid reaction. Let settle

Results —	Before	After
Sp Gr	1.217	1.330
Grms Ni per Liter	59.43	101.504

or not twice as much Ni

also large quantity of Iron in sol. and free acid

Mixture was rather thin grain, but settled clear over night.

If mixture had been heated, I believe it would have settled easier, extracted more Ni, and would have been lower in free acid and Fe.

Jan. 29/20

contin from p-20

To make $NiSO_4$ Sol.
free of Foreign Metals & Chlorine

Cand $NiSO_4 + 6H_2O$ from Bbl.
Water " Tap.

Sol. contains free H_2SO_4
Fe Cu
probably other metals
also dirt etc.

Added very small amount of $FeSO_4$ xtals — heat to boiling — Oxidize Fe with $BaCO_3$; (excess O shown by KI & Starch) neutralize and ppt. Fe & about 3% of the Ni with $BaCO_3$ *($BaCO_3$ is cheaper than $Ba(OH)_2$ and has the advantage also, that the CO_2 given off, will help drive out the free O) — $BaCO_3$ did not take out last traces of Fe, ~~[illegible]~~ but all H_2S groups were absent. — To ppt last traces

* $BaCO_3$ will not ppt. Ni — but it will neutralize ~~[illegible]~~ all free acid, and ppt most of Fe, — $Ba(OH)_2$ must be used to ppt $Ni(OH)_2$ & last trace Fe

Contin on p-24

Cassiter.

Incidentally all Pb - Cu - Al. P - As - Sb - Bi - Se - Te etc ppts also.

Filtered :-
Solution neutral, but showed Chlorine,
Added $AgNO_3$, in Sl. excess To collect ppt of AgCl and ppt excess Silver, used in

553 (1) Excess $BaCO_3$ and filtered clear sol. = 1.37 Sp Gr.

554 (2) Excess BaS and filtered clear sol = 1.30 Sp. Gr. after 1 hr.

(1) Showed dark & laminated.
(2) " " & " "

Showed as above for about 4 deposits, & stopped exp.

#1) Showed black sediment in bottom acide of Ni etc.

#2) Mr. Edison added $NiSO_4$ and tried again deposit was finner, but dark in color.

Cassiter.

Jan 30/70 Carried from p. 21

3 ~~29~~* C.p. Tilt N½ for mark

Feb. 3/20

Sample of Cuprous Chloride
from Storage Battery Plant

30. 0.730
(15.6938 Iron
14.3792

30 : 0.73
23.797
6.776

Moist. :- 43.45 %

Used 8.1037 gram (dry)
1/1 = 0.368 gram Insol. HCl Insol :- 2.57 %

Water Sol :-	1/10 = 10.6	cc Hypo	= Cu	4.61
Neutral (	1/10 = 1.75	cc Key	= Ni	1.30
Neutral)	1/10 = 0.8	cc $K_2Cr_2O_7$	= Fe	0.31
	1/10 = 2.8	cc $KMnO_4$	= CaO	0.54
	1/10 = 0.296	gm. $BaCl_2$	= SO_3	7.09
	1/10 - 1/5 = 0.5	cc $AgNO_3$	= Cl	2.43
HNO_3 Sol.	1/10 8.9	cc Hypo	Cu	3.87
+ HCl Sol.	1/10 0.3	cc Key	Ni	0.24
	1/10 0.2	cc $K_2Cr_2O_7$	Fe	0.08
	1/10 = 53.0	cc $KMnO_4$	CaO	10.29
	1/10 0.610	gm $BaCl_2$	SO_3	14.61
	1/10 - 1/5 = 1.0	cc $AgNO_3$	Cl	0.54

(See page 34)

Feb. 3/20

Sample of Cement Copper
from Storage Battery Plant

31.5500
(12.0460 Iron
20.5040

31 55 00
26.2470
6.3030

Moist. :- 30.76 %

Used 14.201 gram (dry)

Feb. 3/20 40 #per day

Metal Sponge

Sample of ~~Cement~~ ~~Copper~~

from Mr Dinwiddie

38.100 38.100

3 (13.001 33.431

1 4.099 4.669

Moist. 33.11

Used 20.430 grams (dried then ground

Porter Sol.

1/10 = 24.0 cc $AgNO_3$ = Trace 1.82 %

Contained no metal.

1/1 = 0.022 grms Insol = Acid Insol. 0.11 %

1/5 - 1/10 = 15.4 - 20 cc H_2PO = Cu .3347

1/5 - 1/10 = 24.0 - 23.9 cc KCy = Ni .6602

1/5 - 1/10 = 1.0 cc $K_2Cr_2O_7$ Fe_2O_3 .093

1/5 - 1/10 = 16.0680 ~~cc H_2PO_3~~ Co 5.64

15.8817 Cu_2Cl_2 = 15.75

Ag Cl .1863

Moist by dish 2.01 %

Note Spec dried out in Laboratory before analyses. — Did not have very much Moisture originally as it came in Gum-processes Circ.

3/3/20

Continued

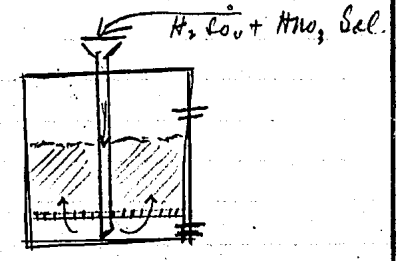
Treatment for 40 # per day Dinwiddie Sponge Metal.

Cannot be prolonged — or the Ni and Cu separated by screening.

Dissolve in acid-proof stone ware crock with dilute H_2SO_4 and just enough HNO_3 or Na_2NO_3 or $Cu(NO_3)_2$ to just neutralize the dissolving.

Then this sol. of $CuSO_4$ + $NiSO_4$ in one of the other Mixing Receivers.

The apparatus something like this :—



3/3/20

But not the Speich.

7/11/20

continued from page 30

Copperas Chloride

Moist			43.45
HCl Insol. #			2.57
	Water Sol.	HNO_3 Sol.	
Cu	4.61	3.87	= 8.48
Ni	1.30	0.24	= 1.54
Zn	0.31	0.08	= 0.39
CaO	0.54	10.29	= 10.83
SO_3	7.09	14.61	= 21.70
Cl	2.43	0.54	= 2.97
Al	Trace	Trace	
O - H_2O & Undetermined			8.07
			Total 100.00

* Insol is practically all SiO_2 } Total 100.00
Trace, organic etc present }

$CaSO_4$ 26.29 %
Moist 43.45 %
HCl Insol (SiO_2) 2.57 %
$CuSO_4$

Continued.

Check on I-Cl

104.686
96.233
6.453 gm 1/5 = 4.30 cc Ag NO_3
= Total Cl 1.-

Examination for other Metals

97.825
82.648
15.184 gr

Feb. 18/20

Extracts from Letter of 7/11/20 from Mr. C. E. Carter & Mr. L. A. Edwin describing by-products of Edwin Strange Battery Depr.

Mr Edwin's Comment :-

Doughty : Investigate these by-products when you get time & get samples & make experiments. Return letter to Mr Meadowcroft when you are through.

Copper Cement A reddish, granular mud, usually containing

Moist. 30. %
Copper 45.
Nickel 3.

production 1200 # per week.

Nickel Iron Hydrate (Mud) A yellow ish-brown sludge, containing about

Moisture 55. %
Nickel 8-9. %
Iron 3. %

also quite a little lime and small amt of Copper.

Production about 8000 # per week.

Cuprous Chloride :- A reddish grey granular mud, containing :-

Moisture :- 30. %
Cu :- 15. %
Ni :- 2. %

also quite a little lime

production est. 1500 # per wk.

Copper & Nickel Shot Scrap

Metallic Scrap.
Copper est 20 %
Nickel " 80 %

production est 300 # per wk.

Nickel Anode Drillings

Metallic Ni Drillings
Ni :- est. 98.5 %

production est 7 # per wk.

Nickel Sludge :

Dark brown mud containing about

Moisture :- 30 %
Nickel :- 50 %
Copper :- 5 %
Iron :- 3 %

Accumulates at about 100 # per wk.

Nickel Carbonate from Sumps. This material, as far as it can be pumped from the sumps with the

Contin.

present equipment, is used in the regular process, without any surplus accumulation.

Copper Carbonate from Sumpo

With increased equipment now installed all Carbonate from Copper Sumps is being turned into Copper Sulphate.

Washed Nickel Sludge from Plating Tanks

Metallic appearing granules

Nickel Contents at 80%

Considerable Fe added present production at 50# per 24 hr.

Nickel & Iron Sludge, Solution settling Tanks.

Dried yellowish brown powder, mostly Amorphous Tempered.

Nickel Contents at 50%

Large Amt. of Fe present

Accumulation of about 250# per 24 hr.

Hydrated Lime from Acetylene Generator.

This material could be collected and stored dry, to permit any Carbide present & decomposed, and the resulting Hydrated lime ~~stored~~ and either sold or used somewhere in the Edison industries.

Feb. 13/19

Quantitative Exp. to extract Nickel from Copper Chloride Mud.

Note (Material may be dry or wet mud.)

Took 200 grms + 200 cc Water

Vol of Mud 125 cc

" " Water 200 cc

act. Vol. of Slurry 335 cc

After adding 100 cc more Water 435

Analyses shows 3.87 % free acid Vol.

= 11.95 grms H_2SO_4 for the 200 gr

= 13.95 " 85.59 % H_2SO_4

Store acid 1.79; Spec. No 13.95 g = 7.8 cc

Added 10.0 cc

After standing 1½ hrs. only large crystals $CuSO_4$ settled as granular sediment. — Top liquor full of finely suspended matter. — Added 100 cc Sharp Water & ~~10 grms Salt~~

After settling over night (15 hrs) settled clear enough.

Filtered on Buchner funnel

Filtrate + Washings 388 cc

~~Added 20 cc [illegible] Cu~~

~~Washings~~

Press Cake = 156 grms. Cake Dry

" " contin. 100 gr = 42

Cuprous Chloride (Contin.)

1/10 = 25.0 cc Hypo = Cu

Cu left in Dust :- % of Total

Note 1/10 = 22.5 cc Hypo

NH_4OH sol. = %

HCl Dissol = none

50 cc Sol

1/10	29	cc Hypo = Cu
1/10	4.4	cc Key = Zn
10 cc	18.7	cc NaOH = Fe as Fe_2O_3
1 cc	6.0	cc $K_2Cr_2O_7$ = Fe
10 cc	3.6	cc $KMnO_4$ = Cao

Feb. 14/20

Preliminary Exp. to Utilizing Cement Copper from Storage Battery, Dry material may be dryer than sample that was analyzed ~~Last Time~~. See analysis.

Took 200 gm + 10 cc H_2SO_4 + 200 cc H_2O (H_2SO_4 same as Cu Cl Exp.)

Vol of Cement Cu 91 cc

Vol " H_2O 200 "

Actual Vol of bath. 300 "

Filtered & used small quantity of Work water —

Filtrate & Washings :- 368 ccs.

Press Cake 146 grams (wet)

" " 100 " (dry)

Placed crucible over B-Burner — grease & organic matter burnt off. — (considerable present) — Oxidized, or at least turned black by readily, & after stirring occasionally for 3 hrs., apparently finished.

Wt. after oxidation = 50 grms.

Large proportion of fines in calcined mat'l

Note: Before doing the above, dry press-cake is divided into 2 equal parts #1 and #2

(Continued)

#1

Treated as above

After oxidation, wt. = 50 grms

Sifted thru' 180 mesh screen

Act wt. of Underside #9.05 grms = [illegible] %

" " " Oversize 0.95 " = [illegible] %

Underside

1/10	gm =	37.2	cc Hypo CuO 50.23 %
	" =	0.8	cc Kly Ni 1.80
50 gr	" =	0.05	cc $K_2Cr_2O_7$ Fe_2O_3 0.08
"	" =	0.4	cc $KMnO_4$ $CaSO_4$ K_2O 0.40

Oversize

0.95	gm =	54.0	cc Hypo
"	" =	—	cc Kly
"	" =	0.1	cc $K_2Cr_2O_7$
"	" =		cc $KMnO_4$

#2

Boiled with xs Na_2CO_3 to convert $CuSO_4$ to $CuCO_3$ — Filtered and washed away sol. Na_2SO_4 — Suspended in water & then dissolved out the $CuCO_3$ with Cl — excess HCl. ($CuCl_2$ also dissolved, look into this as perhaps it might be a good idea to treat at outset with a mixture of HCl & H_2SO_4) Some of ppt. boiled over leading to

(Continued.)

a loss, so that ppt. cannot be finished quantitatively.

Filtrate — washed partially, dried and calcined. — In calcining a great deal of fumes given off ($CuCl_2$)? — Calcined at a comparatively low temp. but the fumes came seemed heavy.

Pass through 180 mesh screen

{ Underside = 26.0 gm
{ Oversize = 0.01 "

Solution & Titrations

			gm per l.
50 cc	1/10 =	11.0	cc Hypo = Cu
"	1/10 =	3.1	cc Kly = Ni
10 cc	4.3		cc Zook = from H_2SO_4
10 cc	5.7		cc $K_2Cr_2O_7$ = Fe
10 cc	—		cc $KMnO_4$ = CaO from then

See next page for analyses.

Contin.

Underreagent

	1/10 =	46.5	cc HgO	= CuO 72.54%
	=	1.5	cc KCy	Ni 2.69
5.0 gm	=	0.05	cc $K_2Cr_2O_7$	Fe_2O_3 0.08
	=	14.0	cc $KMnO_4$	$CaCO_3$ 14.76

Overreagent

0.21 gram = 14.6 cc HgO
" " = 2.2 cc KCy
" " = 0.2 cc $K_2Cr_2O_7$
" " = 0.05 cc $KMnO_4$

#1 Shot = 0.131 gm Insol
#2 Shot = 0.047 " "

This is principally graphite

Making the differences, as follows in Cu percent, as follows:

#1 Shot	%	$CaCO_3$
#2 Shot	%	" "

#1 Trim 0.022 gram Insol (SiO_2) = ?
#1 " 0.005 gm $Fe_2O_3 + Al_2O_3$ - Al_2O_3
#2 Trim 0.044 gm Insol (SiO_2)?
2 " 0.0045 " $Fe_2O_3 + Al_2O_3$ Al_2O_3

Contin.

	#1 CuO	#2 CuO
CuO	50.23	72.54
Ni		2.69
$CaCO_3$		14.76
Fe_2O_3	0.05	0.05
Al_2O_3		
Insol.		

Feb. 18/20

2 phial samples of Cu O made from Cement Copper (E.S.B.) and given to Mr. Edison

#1

Tried to remove Nickel-flake from Cement, by boiling with dil H_2SO_4 & Na_2CO_3 — Wash free of Col. salts — Filters — Dried — Calcined — to oxidize easily by placing in porcelain table first on gauze & then on triangle. — Pulverized & passed thru 180 mesh.

Result:-

Oversize still contained considerable Nickel flake — Fines was full of $CuSO_4$ & had gray tinge.

#2

Took finest mat'l, that Mr. Edison produced by floating & screening. — Boil with xs Na_2CO_3 — free of sol salts — Treat then with xs HCl to dissolve $CaCO_3$ — Wash — Filter — dry — calcine same as above, ~~& pass thru~~ 160 Mesh Screen

Result:- Fines were black & good quality, but oversize contained ~~likely~~ small amt. of Ni flake.

Feb. 18/20

Cement Copper from E.S.B.

Removed w. sol. salts & then converted $CaSO_4$ to $CaCO_3$ by boiling with sl. xs Na_2CO_3 — Freed of Na_2SO_4 by washing & handed over to Mr. Edison.

Mr. Edison tried by levigation to separate into 3 parts.

Lights. containing bulk of $CuCO_3$
Med. " " " Cu
[redacted] " " " Ni flake

Dried just at room temp =

Lights	14.165 gms	=	14.87 %
Med	54.58 "	=	72.73 %
Heavy	9.303 "	=	12.40 %
Total	75.048		100.00

Not thru' with this

Mch. 3/20

New Method of purifying $NiSO_4$

Took Lunch × Cols. ($NiSO_4$ + 6 Aq.) from Bot.

Heated in Casserole on gauze over B-Burner, it practically dehydrated. — Noticed peculiar odour.

Cooled — Added distilled Water — At first the material disintegrated with separation of Basic Sulphate, but, as temp rose, increased the basic Sulphate went into solution.

After boiling for 1 min, we had sol. rose acid in reaction. — Added pure ammonia, Bot. it, to neutralize — Boiled — This is supposed to ppt. Co. Fe. Al etc. — Filtered — Added 1 drop H_2SO_4 (concn.) to 250 cc. 1.30, Sp. Gr. nearly — Tested it B-Litmus paper.

1st deposit :- Slightly foliated & brittle & dark-looking N.G.

2-3-4 ×s Seemed to be pretty good

Sent specs to Mr Edison

Mch. 9/20

Pure Ni Co

In exp. on previous page noted that kernals were not of uniform size, the larger being blue in centre, and the finer yellowish-green —

Suspecting the large pieces might contain some undecomposed impurity that might spoil deposit I froze up the Cond. x tube to pass 10 Mesh Screen. — These treated above as before in porcelain-dish on gauze over Bunsen Burner

Gas off not only an acid odor but also smelt and looked as tho' organic matter were present and was decomposing. — Some of the kernals actually turned black as though charred.

Run the 4 following tests and send the samples of deposit to Mrs. Edison.

Mch. 9/20

(#1)

Dissolved dehydrated Ni Co, in distilled water by bringing gradually to boil.

Dissolved slowly at first, acted as though full of basic carbonate of Nickel x sol. Red acid reaction which disappeared gradually, as the ppt. dissolved. — Considerable sediment consisting of dirt, sand, & undissolved Ni. ppt. Some of the sediment was black, also black scum on top. — Added Baryta to neutralize x ppt. the Fe & Cu etc. — Settled very readily & filtered quickly — After standing all night a very sl. sediment formed in bottom — Neutral to B. Litmus — clear & next to darkest green in color.

Sp. Gr. 1.312 @ 28 °C.

250 cc used

Deposits every hour.

Nothing added

Sediment in filter was green-grey

1) Sl. polished on edges, better than #2-3 H- (best)
2) G.K. Ppt. did not turn at edges, firm deposit
3) G.K. turned a little at top, color better than 2
4) G.K. Very good
5) " " " turned at one corner?

54

3/9/20

(#2)

Same as #1 up to the point of neutralization.

Added xs of $Ni(OH)_3$ black granular (free of sul. salts) — The free acid was neutralized more slowly than in #1. — Also settles & filters more slowly than #1. — Ppt in filter was almost black, (due to xs of $Ni(OH)_3$) — Filtrate ~~[illegible]~~ of sediment over night. ~~It~~ about the same color as #1 —

Sp. Gr 1.309 @ 28°C

250 cc used

neutral to B-Litmus paper

(Deposits every hr.)

Nothing added

2d

✓

1) 2nd best, better than standard test Ng.
2) Much better, but brittle on edges, (slightly)
3) O.K.
4) O.K. Perfect
5) O.K. "

55

3/9/20

(#3)

Cond. Nickel Sulphate crystals taken from Bot. — Dissolved in city-water, boiled, added excess $NiCO_3$ to neutralize free acid and ppt. Cu & Fe. — Ppt. was pale green & settled very slowly — Filtered most slowly of the set, but gave the clearest filtrate of the set. — Dark green in color after settling over night, not a trace of sediment.

Sp. Gr 1.308 @ 28°C.

250 cc used

neutral to B-Litmus paper

Deposit every hr.

Nothing added.

2d

✓

1) Very poor, warped, brittle, foliated & dark colored.
2) Much better, but turned at edges & brittle at edge
3) Very good, but turned at corners & brittle
4) Better than 3, but practically O.K.
5) Perfect.

3/9/20

(#4) Standards

Standard Sol.

Made some time ago, as follows
Comm. $NiSO_4 + 6H_2O$ from Bsc.
(contains dirt, free acid, Fe & Cu etc)
Dissolve city water — ~~[illegible]~~
ppt. Cu with BaS — Filtered — Peroxidized Fe with BaO_2 & ppt. Fe & neutralized free acid with $NiCO_3$ — Filtered — Concentrate & Crystallize — Dissolve in Distilled Water (smallest amount of hot) & Recrystallize.
2nd Crystals dissolved in distilled water & made sol.

Sp. Gr. 1.295 @ 28 °C.

Pale green & clear
neutral to B-Litmus paper
nothing added

1) 3rd Rec. Good Color, but bubble & flakes on edge.
2) O.K.
3) Perfect
4) "
5) "

3/15/20

Exp. to find what % Ni is lost in ppt. when $NiSO_4$ liquor is freed of $CuSO_4$ by means of BaS liquor

After ppting the Cu from Ni. Salts Sol. (which was made in Cu.) the black ppt. was filtered onto paper — washed free of Ni-Sol. Salts — dried and assayed :-

1.0 gram = 0.649 gm $BaSO_4$

$BaSO_4$ 64.90 % as Key
1.0 gm $\frac{}{3.8}$
$= NiS$ — 5.18 %

1.0 gm 17.25 as Hypo
$= CuS$ — 16.44 %

.649 % $BaSO_4$ should = 26.59 %.
but Fe, dirt, moisture etc. also present

3/15/20

#1 Exp. To find if a ppt of $BaSO_4 + Ni(OH)_2$ could be used for neutralizing ~~[illegible]~~ and strength during spent Nickel electrolytes.

If this could be used, it has the advantage that it need not be free of Na. Sol. Salts, & will also filter better, and the neutralized electrolyte would also filter better.

Exp. was also run to see if this product could be made from impure solutions of $NiSO_4$.

Took 250 cc Stand. Nitro. Sol. ~~[illegible]~~ purified & doubly X'tallized — added 5 cc conc. H_2SO_4, neutralized this with a pinch, made as follows:— 4/1

~~[illegible]~~ Took some of Calc's stand Sol. (Contains free acetic, also large quantity of Ca also some Fe & Al) — added sl xs $BaOH_2$, & filtered. (filters bad) — Boiled filtrate & added xs $BaOH_2$ and

Contin.

(filtered on Brückner funnel — Filters very easily but contained xs $BaOH$ in the precipitate.)

Added this ppt to acidulated Ni Electrolyte ~~[illegible]~~ (hot) until free acid is neutralized. (some sl xs) — Went into Sol. very readily, — also settles quite readily, but passed thru filter paper at first. Refiltering gave clear dark Sol.

250 cc for test
Nothing added
Sp. Gr. 1.235 at Room Temp.
Started at 9 0 7 m.

4/9/20

1) Brittley flaked away from cathode.
2) Pitted around edges, dark in colors.
3) OK perfect
4) " "
5) " "

Contin from 58

Contin

Exp #2

Same procedure as Exp #1. Last part made in follows.

Concl. TiO_2 treated by Ba and $TiCO_3$ — ~~[illegible]~~ settled. — Sol excess $Ba(OH)_2$ added — filtered. — Most of Ti ppt from filtrate ~~[illegible]~~ by means of $Ba(OH)_2$. —

Settled readily, about half of the process and thrice passed filter twice. — Filtrate dark green & clear.

Sp Gr 1.298 @ 23 °C.

20 drops H_2SO_4 added to the rest and shaken neutral to litmus & M.O.

{ $TiSO_4$ in Pyrex Cake — Filtered } { easily in Bunsen funnel.

1, 2, 3, 4, 5 } All about same as Exp #1

(Contin.)

Exp #3

Suspecting that the poor results obtained in preceding two exp. was not due to Barium, proceeded as follows:—

Sol. of $TiSO_4$ that had been freed of Cu - Fe - As acid by means of BaS, $TiCO_3$ & filtration. — Crystallized twice, separating from mother liq by suction filter etc, and dissolving in distilled water. — Comparatively dilute sol. Was brought to boil and clear $Ba(OH)_2$ sol added with stirring — Leave sl - excess of $TiSO_4$ in solution. Filter directly — Filtered easily & quickly. — Did not work — ~~[illegible]~~ $TiSO_4$ was present in the ppt — Used directly —

Took solution from #2 & 3 Exp. (reported on 3/4/20) after 5 deposits of Ti taken from them

next page

Caustic.

For description of this sol. was made, See exp. pages 54 & 55

Sol. contained free alkali as NaOH as Na2O

2.5 cc = 5.7
= ——
= free H_2SO_4 :- 5.7 grm per L.

Strength of Sample
γ = Na2O. as Na2O.
γ = Na2O.
γ = Na(OH)2 as KOH
H_2O by diff.

For Comparison:-
Na(OH)2 made by regular method
γ as KOH
γ = Na(OH)2
H_2O by diff.

Brought mix 1/2 vol/2 of electrolyte from #2 × #3 Exp. to boil and added pumice directly until neutral by M-O & B-Ph mixed paper.

See next page

Caustic.

(Note Both of these sol's gave perfect deposits and γ = 2.)

Pumice mixed into sol. very easily — When neutral allowed to settle for a few minutes.

Settled readily & filtered easily (the first few cc were a little cloudy but by pouring through filter again it came clear.

Sol. clear & dark green in color
250 cc used
Added just enough H_2SO_4 to turn B-P paper red.
Sp. Gr. 1.32 @ 90 Fah.
Started at 90 Fah.

4/7/20

1) Perfect
2) "
3) "
4) "
5) "

Samples of these Exp. Analyzed by Mr. Edison 4/7/20

This part with same time, distilled water etc. easily filt.-pressed & sol. easily filtered.

3/24/20

4/7

Finding a solvent for Din middlin Nickel Sponge.

Wondered if $CuCl_2$ from Cuprous Chloride recovery caused the trouble.

Did not work so well as ppt of $CuCl$ was formed.

Tried H_2SO_4 in presence of the Chloride of Copper Cinder

Strong evolution of Hydrogen & Nickel seemed to be slightly dissolved, leaving Cu as Cement.

Tried an ounce of Tinman's fine off cuttings Tin plate, Tin Lead of dilute H_2SO_4 with small quantity of HCl, dissolved it very rapidly leaving Copper behind as Cement — I then added a little of Din middlin Sponge & noticed that H was again evolved...

As it was proven that a mixture of H_2SO_4 & HCl would dissolve metallic Tin coming from metallic Copper Cinder it occurred to a segment of Nickel plates

(Contin.)

Copper Disc. Nickel.

Dissolved very readily but also showed small amount of Cu in Sol.

Sample Marked #1 Sent

This reminded me of your statement that the Nickel plate would be dissolved from this material by galvanic action, a dilute H_2SO_4 alone.

Sample #7 Sent also was made by mixing of that dilute H_2SO_4

verifying Mr. Edison's statement

4/7

4/3/20

Assay for Mr. Smith
Cleveland Potash Co.
Marked 33%

28.8455
19.0713
9.7744

= cc N/10 H_2SO_4 for MeO.
" " " Phenol.

= J.U. as KOH 30.57 %
act. " 29.07 %
K_2CO_3 1.78 %

Spls of $MnCO_3$ Sol. that will likely be furnished by Moore

4/9/20

Spls of Copper Oxide? from George Hunt 4/8/20

Spls marked 2.00 Pm & 4.00 Pm 3/27/20
had to be filtered - Washed & dried

1) Spl marked 2.00 Pm. 3/27/20
0.4 gm = 40.15 cc Hypo
Cu = 63.59 %
= Cu_2O CuO

2) Spl marked 4.00 Pm. 3/27/20
0.4 gr 41.8 cc Hypo
= Cu = 65.40 %

3) Spl marked Lime
0.4 gm 40.2 cc Hypo
= Cu 6 Sol %

Note These are all too low for either Cu_2O or CuO. Found moisture present
See Page 75

67 4/9/20

Lost Burn

1) Spec of Copper Mine
Primary Battery Dir
Silver Lake
Spec lost moist. standard

Took 50.0 grms.
45.1
4.9
Moist. :- 9.80 %

45.1 gr 1/20 - 1/10 = 23.75 cc Hypo
- Cu 60.18 %
1/20 - 1/10 3.5 cc $K_4Fe(CN)_6$
Zn 9.50 %
1/20 = 3.8 cc $K_2Cr_2O_7$
= Fe 0.85 %.

✓

68 4/9/20

Lost Burn

2) Spec of Zinc Mine
Primary Battery Dir.
Silver Lake
Spec lost some moist
High in Copper.
Found pieces of CuO plates
(Spec in bottle)

Took 50.0 grms.
46.8
3.2
= Moist :- 6.40

46.8 g 1/20 - 1/10 6.20 cc Hypo
Cu :- 15.71 %
1/20 - 1/10 = 30.0 cc $K_4Fe(CN)_6$
16.45.90 = Zn :- 24.40 %
1/20 = 16.4330
$PbCrO_4$ 0.0260 Pb 0.67 %
1/20 - 2.45 cc $K_2Cr_2O_7$
= Fe 0.55 %

Checkmay
Zn 1/20 = 64.0 cc $K_4Fe(CN)_6$ = Zn % 64.25
" " = 12.5 cc Hypo . Cu 15.71

✓

69 4/9/20 Lost Bar.

3) Spls of Copper Floor Scrapings
Primary Battery Dir.
Silver Lake
(Spls Lost Moisture Already)

Shot 60 gr. Frock 2.00
Frac. 860 " 43.00
46.00 gm
45.8
Moist :- 0.43 %

45.8 gm 1/20-1/10 27.4 cc Hypo
= Cu :- 15.47
1/20-1/10 - 1.6 cc $K_2Fe(CN)_6$
gm :- 3.26 %
1/20 = 3.75 cc $K_2Cr_2O_7$
= Fe 0.89 %

70 4/9/20 Lost Bar.

4) #11 R.C. Copper Wire
from Electrolytic Elements
Primary Battery Dir.
Silver Lake

B.B. 14.89 gm } This is an
A.B. 14.037 " } average piece

14.037 gm 1/10-1/10 = 27.0 - 27.0 cc Hypo
= Cu :- 70.10 - 70.13 B.B.
Cu :- 99.33 - 99.31 A.B.

71

4/9/20

5/ Sple of Regular Zinc Brass
from Premium Bolting Div.
Silver Lake
Sple lost Moist. by Heating

Zinc	300.0		300.0
Shot	25.5	Tare	25.5
			275.5
			275.5

= Moist. in wm

1/20 - 1/10 50.5 cc $K_4Fe(CN)_6$

= Zn 91.36

1/20 - 1/10 0.25 cc Hypo

= Cu 0.57 %

1/20 - 1/10 0.15 cc $K_2Cr_2O_7$

= Fe 0.30 %

Some Insol.

72

4/9/20

5) Sple of Reclaimed Zinc Brass
from Premium Bolting Div.
Salem Lake N.J.

Zinc	545.0		545.0
Shot	79.2	Tare	79.2
			62.42
Sple lost Moist by Standing			62.42
			0.00

Moist. in wm

1/20 - 1/10 53.5 cc $K_4Fe(CN)_6$

Zn 96.05

1/20 - 1/10 2.25 cc Hypo

= Cu 4.56 %

1/20 - 1/10 0.05 cc $K_2Cr_2O_7$

= Fe 0.09 %

[redacted] Insol.

4/9/20

7) Sper of Zinc & Oxide Sweepings from Primary Rolling Div Salem Lake Zn

Spec Sort Moist. by Chand &

Fines	350.0 gr		17.5
Short	210.0 "	Tare	10.5
Paper, Wood etc.	12.0 "		0.6
			28.6
			28.6
Moisture — None			0.0

1/20 − 1/10 = 15.0 as K4Fe(CN)6
= Zn 55.5

1/20 − 1/10 = 55.50 as Hg(NO3)2
= Cu ?

1/20 − 1/10 = 0.25 as K2Cr2O7
= Fe 0.97

24

Considerable Green & Frost.

Check Assay as opposite

Fines	350.	Tare	17.5
Short	210.		10.5
Paper, Wood etc.	12.		0.6
			28.6

Moist. None

1/20 − 1/10 = 15.9 as K4Fe(CN)6
= Zn 55.8

1/20 − 1/10 = 6.0 as Hg(NO3)2
= Cu 21.09 %

Fe 0.97 %

24

4/26/20 →

Contin. from page 66

Copper Oxide Spec? from G. Hoard

1) made 2 P.M. 3/27 Calcium Hypochlorite

8.1945
5.7656

8.1945
8.2333

gained wt.

.4699 gm dry
1/4 = 55.5 cc Hypo
= CuO 71.33%
CuO by diff
NaClO

2) made 4.00 P.M. 3/27 Sodium Hypochlorite

5.6223
6.5440

8.6223
8.6447

gained wt.

.1007 gm dry
1/4 = 47.0 cc Hypo
= CuO = 70.97%
CuO
Cu(ClO)

3) made Lime

8.1153
6.0540

8.1153
8.1550

.101 gm dry
1/4 = 52.0 cc Hypo
= CuO 78.71
1/4 = 16.80 cc Hypo
= CuO 20.88 by diff

gained wt.
1.5 gm = 1.5 cc $KMnO_4$ Lime as CaO = 0.41% dirty
Cu(ClO)

Contin.

Note

Attempts to determine the Manganese, but Spec were further oxidized, and gained wt.

Calculate results on the calcined Spec.

All Contained Metallic Copper

4/27/20

Small Spln of Steel Turbine
from Mr. Bennie 4/26/20

Complained of by Mr. Warner
(Used all of Sample)

(4.1158 grams used)

1/1 = 0.7 cc Hypo = Cu 0.11 %
1/4 .54 cc $K_2Cr_2O_7$ = Fe 0.73 %
1/20 = 19.05 – 19.00 cc HCl
= Ni 99.05 – 98.79 %

as, Cr, Pb, al, none
SiO_2 trace

27

Small Spln Nickel Anode
from Mr. Warner
7.7777 grams. (Used all of Spln)

1/1 - 13.5 cc Hypo 100 = Cu 1.09 %
1/1 - 13.0 cc $K_2Cr_2O_7$ Fe 1.09 %
1/10 - 1/10 = 14.97 - 14.90 cc HCl Ni 97.67 - 97.59

	I	II	aver
Ni	97.67	97.46	97.59
Cu			1.09
Fe			1.09
SiO_2			.26
C			
			100

5/18/20

Small Spln of Zn-Mn Bellows
from Mr. Edmund Smith.
4/18/20

Marked #1

	I	II
Wt of Sample	0.5 gm 7/10	0.5 gm 7/10
cc $KMnO_4$	0.4	0.45
% Mn	0.33 %	0.38 %

aver = 0.36 %

Marked 4% Mn 5/7/20

	I	II
Wt. of Spln	0.5 gm 7/10	0.5 gm 7/10
cc $KMnO_4$	6.4	6.5
% Mn	5.23 %	5.31 %

5/19/20 aver. = 5.27 %

5/25/20

Check assay. Ni Electrolyte from A.P. Dept, which worked poorly (130 A. 5/20/20
10.0 cc = 0.4 cc Hypo = 0.025 gm Cu per liter.
Took portion dry boiled with
Sl. xs Br_2 & filtered & electrolyzed after
addition of 3 drops concen. H_2SO_4.

See next page

5/21/20

3 Small Specs of Zn-Mn Alloy
from Mr. Smith 5/20/20

Mark M I 5/20/20
1.0 grams 1/5
= 1.0 — 1.0 cc KMnO.
= Mn = 0.818 % — do

Mark M III 5/20/20
1.0 gm 1/5
= 1.0 — 1.0 cc KMnO.
= Mn 0.818 — do

Mark Mn 5 5/20/20
1.0 gram 1/5
= 1.0 — 1.0 cc KMnO. 6
= Mn = 0.818 — do

---

contin from page
Result of 1 Mn. Mass.
1) Very bad, foliated, curled up & crumbled.
2) Color & pliability very good, but split at edges.
3) Color good, edges good, but pulled away at top.
4) Pulled away at one corner; thinner in center than edges.
{ Probably the most round. Those have perfect. }

5/27/20

Nickel Electrolyte 7.30 Hrs. 5/27/20
100 cc = 0.1 cc Hypo Sol = Cu 0.0063 gram per L.

Check Assay on Dietzy
Nickel Anode drillings 5/25/20
10.0 gram 1/1 = 4.6 cc Hypo = Cu
10.0 " 1/1 = g Fe_2O_3 + Al_2O_3
10.0 " 1/1 = 9.7 cc $K_2Cr_2O_7$ = Fe
Al none
100 " 1/1 0.007 g CiO_2 SiO_2

Zn-Mn Alloys from Mr. Smith
Mark M 6 5/27/20
20 g 1/5 = 1.1 — do
cc KMnO.
= Mn = 0.45 — do.

Mark M 10 5/24/20
20 g 1/5 1.8 — do cc KMnO.
= Mn 0.74 — do 6/12

Mark M VII 5/26/20
20 g 1/5 = 3.1 — do cc KMnO.
= Mn 1.26 — do
Mark M VIII 5/27/20
20 g 1/5 = 0.6 — do cc KMnO.
= Mn = 0.25 — do

May 22/20

Spec of Iron used at Science Lab for Working $FeSO_4$ and Iron Alg-Alg deriv in

Dissolved in Dil H_2SO_4 — added concentrated $MnSO_4$ — When neutral, filtered. — Concen to crystallizing point and added small amt HCl, — treated — filter — agitated with small amt of dilute HCl & filtered again.

Products :-

1) gm X Tolo
2) cc M-Liq
3) Wash Liquor

1) X Tolo

10.0 gm	1/10	34.7	cc $K_2Cr_2O_7$ = $FeCr_2O_7$ + H_2O
10.0 "	1/10	0.4	cc NaOH = free H_2SO_4
25.0 "	1/5		cc $KMnO_4$ = Mn

2) M Liquor

10 cc	1/1	25.0	cc NaOH = free H_2SO_4
10 cc	1/10	16.0	cc $K_2Cr_2O_7$ = $FeSO_4$ + Mg
10 cc	1/5 =		cc $KMnO_4$ = Mn

Continued.

3) Wash Liq.

10 cc	1/1 =	16.0	cc NaOH
10 cc	1/10 =	16.6	cc $K_2Cr_2O_7$
20 cc	1/5 =		cc $KMnO_4$

5/26/20

Exp #1

This so. Electrolyte that was
skimming head. Spec. from Filter
press 100 Pm 5/26
just as it was
Coloring
Nothing added

Note From #1 on deposits were good quality
but showed tendency to pull away at corners

1 Dark looking, split edge, curled, & pulled away
2 Much better on edges, but pulled away
3 Better still, but turned at corners
4 Slightly better than 3
5 " " " 4
6 " worse " 5
7 " " " 6
8 Perfect

5/26/20

Exp #2

Do

Sol. was over what was necessary
to prep. Cu. of Brass Rod.
added in cold.
& Filtered through paper
Filtered easily
Filtrate clear
Nothing added

Split & brittle at edges & pulled away.
2 Perfect
3 "
4 "
5 "
6
7
8

5/26/20

Exp # 3

Do

Added small amt of $Ba(OH)_2$ sol in cold so that some ppt was ppted, at room temp. Filter thru paper

Had to pass 1st filtrate through paper again, second filtrate became clear.

Nothing added

Foamed at anode, more than #1 & 2 a thick ppt, forming a sediment rather heavy, which settled at bottom a greenish color

Note all held firm at edges & corners

1 Very bad, laminated, brittle, but held firm at corners

2 Slightly better than 1 " " " "

3 Sl. better than 2, but split lengthwise

4 Big improvement, but brittle at edges

5 Very much better, but brittle edge.

6 Perfect

7 "

8 "

Contin.

Note When I attempted to remove 1st three plates, it was necessary to scrape, when all of it crumbled it was so brittle

Think this is due to basic condition of electrolyte Examine

5/27/20

Suspected that either the cloudiness due to poor filtration, or lack of water in causing poor deposits.

A

In Electrolyte from Disc Record Dept. Without Filtration.

Peculiar behavior of turning up at edges.

Note All pulled away from cathode showing thin film that cannot pack.

1 Brittle & laminates at edges
2 Much better, but split & pulled away.
3 Good but curled at edges
4 " " " "
5 } Each one a little better
6 } right on to #8 but none
7 } perfect, even #8 showing thin film
8 } of Ni under regular deposit, which tend pulls away

5/27/20

B

Duplicate of #1 + 5 cc dil Bad leg to 250 cc Electr. w filtered, this was simply 8 ppt. colloidal matter.

1 Brittle & laminates at edges tends at corners
2 Much better, but brittle, pulled away at edges
3 Better still but curled at lower corners
4 Perfect.
5 Good Quality, but pulled away & plated underneath.
6 About same as 5, but not plated so much underneath
7 Perfect
8 "

5/27/20

#6

Duplicate of #1 + 2.5 gm
Ni acetate x tbls. & filtered

Note - All showed film of Ni underneath

1 Very good, but bottom slightly off edge
2 Worse than 1, pushed under mesh
3 Pulled away at top
4 Better
5 Firm
6
7 } All good quality but not perfect
8

Note On next

of lack of time, space etc. had to throw aside a lot of partially finished expers. so as to finish up Aprod.

Expts etc
Mn from Fe etc.
Sn " " etc.

Analyses of

6/4/20

1st Crop of Crude $NiSO_4$
from Silver Lake Salvage

Test grm

Trial.
cc 7 $NaOH$ = free H_2SO_4
cc Hypo = Cu
Fe_2O_3
Ni
= $NiSO_4$

Took for Dubly X Tablegiving
6.20 grms = 5.10 cc

Added 200 cc city water
Vol. still 5.10 cc

Heated
At 45 °C 3/5 of X tals dissolved
" 70 °C 4/5 " " "
but salting out rapidly
Added 200 cc more city water
At 90 °C practically all in sol.
Sol. seemed too concen.

so added 100 cc more City Water
Total Water 500 cc

Reaction acid, $CaCO_3$ and black specks etc in suspension
Added Nickel Hydrate (containing Copper etc E.S.B.) until neutral Methyl Orange

Note { Used all of the Spls I had for analysis of Nickel Hydrate
Recovered at Stan. Battery

After showing neutral, concen to

June 17/20

Spls of Rolled Mn Anode
from Machine Shop
to be used in Chem. Room 6/17/20
Recd from Mr L. Colt

24.35 grams taken

1/1 = 0.065 grams Insol
= Insol. =

1/1 11.5980
11.5300
0.0680

1/1 = SiO_2 ——— gr Al_2O_3

1/1 = 28.7 Al_2O_3 trace as $K_2Cr_2O_7$
= Fe_2O_3

1/1 5.0 as Hypo
= Cu 0.13 %

Spls of Zn - Mn alloy from Mr. Smith
Mark M XIV 5/29/20
20 grm = 1.5 - 1.0 as $KMnO_4$
= Mn =

6/25/20

Sple of White Sediment
scraped from inside surface
of Electrolyte Circulating Tank
(Examined for Mr Edison

Appearance from White to gray
Light & Fluffy
upon Heating Gives SO_2 fumes
showing presence of Sulphites
also give off water.
Burns or strongly fumes
Carbonaceous matter

15.10 g	15.10 g
17.86 g	14.786
2.740	.316

Loss on Heating :- 14.10 %

Principally Water :-
Some Sulphur :-
also traces of Rubber :-

1/50 16.3895 gm = 1/1
16.3070
.0825
Pb CO_4 36.80

= 1.80 as Hydr
= $CuSO_4$ 12.80 %

1/1 = 1.192 gm Insol {acid & Ann acetate}

Contin.

1.153 gm — 59.9510
59.9000
.0511

Pb Sol + H_2O

1.153 = 1/1 gm H_2SO_4 x Am Acid Insol
→ 0.805 gm

Loss on Heating	14.10 %
Pb SO_4	36.80 %
Cu SO_4	12.80 %
SiO_2	25.00 % about
Al_2O_3	07.00 % "
Ba SO_4 "	none
free H_2SO_4	present.

Principally Water & Sl. Tonnage SO_2

Traces of Fe
no Ag or Sb.
Pb no Bismuth
" no Calcium
no Ni or Co.

6/30/20

Sple of Scrap from Pockets
from Storage Battery
Sple from Mr Schell
1.554 grams used

1/10 = 25.3 - 00 cc $K_2Cr_2O_7$
= Fe 1 - 90.7 - 00 90.7
1/10 = 1.5 - 1.55 cc KCy
= Ni 1 - 9.06 - 9.38 9.23

Actual Test

4.982	grams	Original	100.00%
2.388	"	Residue	47.93%
2.594	"	In Sol.	52.07%

1/10 Ash =	—	cc $K_2Cr_2O_7$ = Fe	97.64 by diff
1/10 " =	1.3	" KCy - Ni	2.36
1/10 Res	34.2 -	cc $K_2Cr_2O_7$ - Fe	83.95
1/10 Res	4.00 - 00	cc KCy - Ni	15.75

on
Sle ~~Test~~ page

6/30/20

Spln of Copper Matte
Shipped from Stanaya Basin
to Bulbach S & R Co

Spln Marked Reserve
D 397 6/21/20
H_2O :- 17.00 %
Suspect that Bulbach's assay
was low. and assayed
spln taken at Bulbach by Mr
Woodhull. & assayed at Mr Edwards
Laboratory

Took 20.0 gms.

1/20 = 1.0 gms
= 40.0 — 40.8 cc. Hypo
= Cu :- 25.58 — 25.46 %
Ave 25.52 %
= Cu 21.18 % Wet Basis

7/8/20

7/7/20

Spln of Copper Oxide that was
to be used for Hartz Exp.
Supposed to be best turned out
at Clear Lake.
Given by G. Hartz 7/9/20

CuO :- large proportion
ZnO :- Great deal present

Spln of Ore
from Mr. Caleb G. Collins
Kelly's Ford, Va.
Supposed to Contain :-
Co 4 1/2 %
Ni 4 %
Ag 8 oz.
Check qualitatively.
100 gms =

~~200 gm~~ Qualitative examination
seems to verify the above
figures.

See next page for quantitative
examination.

101

2.0 gm = 0.068 do g Al_2O_3 = Al_2O_3 3.43 % Total including Insol

~~Sample of Ore~~

July 9/20

1.0 g 1/5 = 80-80 ¢ = Mn = 6.54 do = MnO_2 = 9.40

Sample of Ore
from Mr Caleb G Collins
Kelly's Ford. Va

Supposed to contain

Ni	4.0 %	0.5 gm and Assay of Insol for SiO_2	
Co	4.5 %	12.0240	
Ag	8 oz.	11.5653	
		.4587	
		act. SiO_2	91.74% of Insol

2.0 gm = 1.483 – 1.486 gm Insol
= Insol. 74.15 – 74.20 – 74.18
= SiO_2 68.03 – 68.07 68.05

2.0 g = 7.3 – do cc $K_2Cr_2O_7$
= Fe_2O_3 29.1 – do

2.0 g gm Borax.
= S Sl. trace

2.0 g 22.0370 – 22.0225 = .0145 ; 22.2370 – 22.2202 = .0168 Dimethylglyoxime
Ni 0.15 – 0.17 = 0.16 % trace
cc Hg do

5.0 g 3.3
= Cu 0.41 %

2.0 g 0.024 – do g Co_3O_4
= Co 0.88 – 0.88

2.0 gm = g Al_2O_3 = Al_2O_3 %

100 gm = 5.7700
5.7668
.0032 AgCl
Ag = 0.00241 %
7/10 oz.

No Pb-Cu-Sb-Bi
MgO sl trace
Cu trace = Ag = –

See page 108

July 10th 102

Nickel plated pockets used to hold Iron by Hydrogen
Scrap from E.S.B.C.

Salvage Dept can buy 50 gm Tons per Mo @ $10.00

Assay { Fe 90.67 %
Ni 9.23 %

			¢
Nickel	= 48 ¢ per lb.		40
Venetian Red	= 14-16 ¢ " "		12
Sudan	3½-7 ¢ " "		4
Copperas	2.50 per 100 #		2½

2240 # × .0923 = 207 # @ # 0.40 = # 82.80
2240 # × .9067 × 1.4298 = 2904 # @ # .12 348.48
2240 # × .9067 × 1.4298 / 0.6 = 4840 # @ # .04 193.60
2240 # × .9067 × 4.9786 = 10112 # @ # .025 252.80
82.80 × 50 = # 4140. # 348.48 × 50 = # 17424.00
193.60 × 50 = # 9680 ; # 252.80 × 50 = # 12640.00
2034 # Fe = 3566 # H_2SO_4 = 3834 # 93 %
@ 16.00 per Ton = # 30.67 × 50 = # 1534.00

July 14

Spln of Nickel Electrolyte
from Bldg 27
10,00 P.m. 7/14/20

100 cc = = 7.9 cc Hypo
= Cu :- 0.181 grams per liter

July 20/20

Check assay
Spln of Cu Co., 7 cc. from Mr Eckman Lobby, Spln from Mr McMullen 12.00 M 7/20/20

35 cc. 12.0 cc Noon sol
= per H_2SO_4 :- 10.03 grams per liter

Nickel Electrolyte A.M. 7/20/20

100 cc. 21.5 cc Hypo = per acid as H_2SO_4 :- 1.032 grams per liter

100 cc of cc Hypo = Cu :- 0.0817 grams per liter

July 20/20

3 Splns of Copper Solution from Mr McMullen 2.00 P.M. 7/20/20

Spln No	Cc taken	cc Noon	H_2SO_4 Grams per Liter
27	50	51.3	30.01
27B	55	48.0	25.53
24	55	57.3	30.46

July 20/20

1 of Sample of Slurry from Tank 2 at Silver Lake, to be decanted
1170.0 gm
510.
660. wet = 425 cc cc Total
Clear sol = 115 cc = % of vol.
Insol. residue - 435 gm (wet).
10. Ag. = cc Kcy = Ni :- gm
34.971
26.001 31.978
Minus :-

4.9 gm wet = 5.7 cc Key = Ni See next page A'

July. 21/20

Soln of Ni Electrolyte in 1 liter cylinder from Mr Edison 7/21/20 Noon

100 cc = 0.25 cc $Na_2S_2O_3$
= Cr 1 - 0.0156 grams per liter

✓

Spent Copper Solution from Mr Muller 7/21/20 3.30 P.M.

55 cc = 31.5 cc NaOH
= free H_2SO_4 1 - 16.75 grams per liter

✓

Copper Sol. McMuller 3.45 7/22/20
350 cc = 18 cc NaOH
= free H_2SO_4 1 - 15.04 grams per l.

✓

#1

Water Sol.

2/10 =		cc NaOH = free H_2SO_4
1/10 =	3.5	cc $Na_2S_2O_3$
1/10 =	4.0	cc $K_2Cr_2O_7$ = Fe_2O_3
1/10 - 1/10	10.4	cc KCy = Ni

July 28/20

Spec of $NiSO_4$ Liquor in Tank #3, Silver Lake
(Supposed to be ready for crystallization)

35 cc = 180 cc NaOH
= free H_2SO_4 150.4 grams per liter
= 9.4 # per cu ft.

Tank 5.5' dia x 5.5' deep
= 1173 # free H_2SO_4 in Tank

Silver to crystallize

Y Tues 10. gm = 14.0 cc NaOH
= free H_2SO_4 4.10 %

M.L. 5.0 cc = 36.5 cc NaOH
= free H_2SO_4 ≈14.0 grams per liter

July 30/20

Spec of $CuSO_4$ Electrolyte from circulation
Bldg 24 7/28/20

20 cc = 5 cc Kly Sol
= Ni 2.675 grams per liter
= $NiSO_4$ 7.049 " " "
= $NiSO_4 + 6H_2O$ 11.978 " " "

5.0 cc = 50.0 cc Hy 100
= Cu 1 - 62.40 grams per liter

Ni = 4.11 % of the Cu + Ni contents

Check on Cobalt Ore (page 101)

20 gm = 59.9220
59.9025
0.0195 by electrolysis
0.97 - 0.95 % Co

Check on Ag in 25.0 gm showed faint trace AgCl

Aug. 4/20

Spec of Brack water
taken by Mr. L. GILLEM
1000 Am. 7/21/20

Looked great deal warmer on lower tiers
— Seized up flow and took as near an average as was possibly could.

1000 cc = Co as KCl
= Ni = 0.05646 grams per L.
1000 cc = Cu as H_2S
= Cu = 0.03687 grams per L.
= $NiSO_4 + 7H_2O$ 0.01684 per cent for
= $CuSO_4 + 5H_2O$ 0.00903 " " "

Aug 4/20

Took MnO_2 crude x tals from Silver Lake Recovery Plant. — Instead of neutralizing with crude NiOH (containing Cu etc) from E.S.B. Co, used (1) carbonate of lime, filtered & x tallized and in (2) used Ni & Fe hydr until the Cu showed in sol. filtered & x tallized
— These x tals are to be x tallized again before considered finished
1) 10.0 gm = 6.0 cc $KMnO_4$ = $CaSO_4$
2) 10.0 " = 0.7 cc $KMnO_4$ = $CuSO_4$

1/30/20

500# per Day, doubley crystallized purified $NiSO_4$ Crystals

500# production of pure Crystals
= 803# Consumption " Carb. "
per day

Tank #1 or Dissolving Tank.

Lead-lined wooden tank cylindrical, fitted with Lead Steam-coil, paddle, and discharge-pipe

[THIS BOOK WAS USED IN BOTH DIRECTIONS.
THE FOLLOWING PAGES WERE FILMED FROM
THE BACK END FORWARD.]

11.5% Spodumene

6700 - X - 2

Ni + Fe Mud & Reclaim
By-product Pl

Feb. 19/20

Specifications for Caustic Potash
June 20, 1918

Figures obtained by Mr. L. G. H. from Mr Mirick
Taken over phone

Dry on 88 - 92% Basis

~~NaOH~~	Max.	1.5%
K_2CO_3	"	6.5 " or 3.00 to 6.50%
$KClO_3$	"	0.25 "
KCl		1.5 "
K_2SiO_3		0.25 "
K_2SO_4		0.5
$K_2S_2O_3$		nil
Al_2O_3		0.025
Fe_2O_3		0.10
CaO		Trace
MgO		"
KNO_3		"
K_2S		"
Cu		"
Sn		"
Zn		"
Mn		"
Organic Matter		nil
Other Impurities		"

Feb. 14/20

Sample of Caustic Potash
from Sanet-Solvay Co. 7/8/20
Marked Caustic Liquor 45%
Drum # C.P. 72
Spec Reid from Mr Ayer 7/29/20
Assay for KOH - K_2CO_3 - KCl - K_2SO_4
Fe - Al - Mn - S - SiO_2
also Metallic Potash

Took 25 cc 80.7600 Sp. Gr. 1.511
43.3313 @ 28 °C
37.4287
= Sp. Gr. ~~@ 1.1496~~ @ room temp.

1/5 N/10 64.1 - 32.05 cc N_2O_5 Phenol
64.7 - 32.35 cc " Na_2O
= Ta. as KOH 49.87
Act KOH 48.89
K_2CO_3 1.13
1/5 - 1/10 v 3.6 - 18.9 cc $AgNO_3$ = KCl 1.12 %
1/10 - 1/10 16.6637
15.4823 v able
0.7814
K_2PtCl_6
= J - K_2O 40.46 % = KOH 46.96
KOH - K_2CO_3 x KCl as K_2O 42.61 %
Showing that Soda is present
in and equiv. to 1.41 % Na_2O

50 gr = 0.0060 gm Fe_2O_3
= Fe_2O_3 0.0093 %
50 cc = 0.0017 gm Al_2O_3
Al_2O_3 0.0023 %

(contin)

Sp. Gr. 1.511 @ 28 °C.

T.A. as KOH	49.87 %
Act. "	48.89 %
K_2CO_3	1.13 %
KCl	1.12 %
K_2SO_4	none
K_2SO_3	"
K_2S	"
Fe_2O_3	0.0093 %
Al_2O_3	0.0023 %
MnO_3	none
SiO_2	"
CaO	"

KOH - K_2CO_3 x KCl as K_2O 42.61 %
Direct Total K as K_2O 40.46 %
showing that Soda is
also present equiv to Na_2O :- 1.41

A little $Fe(OH)_3$ in suspension which does not settle out readily on account of strength of solution.

Feb. 24/20

Notes from Mr. Edison
on Lithia Extraction

Determine :-

1st Time required to extract about 95% of LiOH, with different sizes of flotation particles.

2nd Speed, or time of agitation with different temperatures.

3rd Minimum amount excess lime required.

4th Minimum thinness of mixture of ore, lime & water.

5th Number of extractions required to get 95% LiOH out.

It requires the mining of 6 Tons of rock to get 1 Ton of good grade ore.

Cost per 1 Ton, in mine = $2.; making good ore cost $12.00

Average contents 4% Li_2O from Spodumene and 3% Li_2O for Lepidolite.

If factory is at mine, Coal = $7.00 — Sand Cost. Lime = $3.00 — Wages $4.50 for 8 hrs. for miners and $4.00 for others.

If shipped to Orange, freight = $12 per ton of 2000 lbs.

Auction.

Wages in Orange $4.00 for 8 hrs.
Lime Comp. $2.50
Lime (Carbonic) $7.00
Coal $6.00

Process (Crude)

1st) Break up ore in crusher to 1" or less.

2nd) Ignite ore to yellow heat, take into cold water to quench (suddenly)

3rd) Dry quenched ore (bone dry)

4th) Grind in ball mill (Hardinge) to very fine powder.

5th) Either blow this very fine ore into settling chambers, one of which should have the float chips, showing under microscope that largest particles are not larger than 1/1000" dia.

Or pass the fine powder from the ball-mill into pointed classifier & work out only the 1/1000" particles. The remaining ore should be dried and again run through ball-mill. Wet ore requires a great amount of screens and powers.

Continued on next page

H.

Caustic.

6th) For Lepidolite & Spodumen, use proper, predetermined quantity of Caustic Lime.

9th) Place proper quantity of Ore and Lime, in Iron tanks, provided with stirrers, which can go very slow — Tanks have a steam — bottom, or coils around outside. The whole tank, top, bottom & sides to be very heavily lagged, to prevent loss of heat as it is under heat over long periods, and loss of coal would be very great if there were more than 10 deg. between atmosphere and outside of lagging — The lagging should be covered over with heavy canvas & tightening steel bands used to secure it in its position.

8th) Use a series of tanks, either withdraw the solution from mix, which may not be possible to any great extent, or withdraw the ore to a filter-press, remove as much as possible and replace in tank, adding fresh water — Use the liquor from filter-press & leach the next tank, removing it also by

Caustic.

filter-press for the 3rd tank and so on until the 1st tank has 85 to 95% of the Lithia removed, and fresh water is strong enough in Li & K salts for manipulations for concentration & purification.

9th) The question to be determined is the amount of Lime per ton of ore required.

10th) The temperature of leaching, (under boiling) — My experiments were on test-plate & only just reached a boil.

11th) The number of tanks — filter-presses & apparatus required for 20 — 30 & 50 tons of ore per day.

For Amblygonite, wooden tanks may be used, if works quick.

Soda Alum, or Sulphate of Aluminia, or Sulphuric Acid can be used.

Should be a little warm.

3/9/20

Sple X

Specimens

Gross Sple – Heated in Clay Xable to red-heat in blacksmiths forge — quench in water. — Pulverized to pass 200 mesh in Steel-Mortar (Brisk).

Part of Sample was put thro' 200 mesh. Test as part of Sples laid around and became contaminated with dirt, iron-rust etc.; the sple. was set aside.

{ Will do something with this later }

3/9/20

Specimens

Took a little more than 2000 grms Specimens, (average) from sack. — Two Sples wt. 1000 gr each.

Sple #1) Broken up to pass 1/4" Screen put in Ball mill for 3 1/2 hrs.
Tot = 1030 grms = 30 grms Wt. = 3%
Pass thru 90 mesh Screen (from Iron Balls?)
Oversize 79.53 gr %
Undersize 950.47 " %
Wt (dry) of 1/500 particles (See page M)

(Sple put away on shelf.)

Sple #2) Pieces 1/4" to 1" — heat gradually to 1900 F. for 2 hrs. and quenched in 1000 cc water (city) — Heated up and boiled violently thro' this quenching — Water was boiled down & examined.

Result :- Found Li and Al in solution.

The rock was then dried — broken to pass 1/4" Screen & placed in ball-mill for 3 hrs.

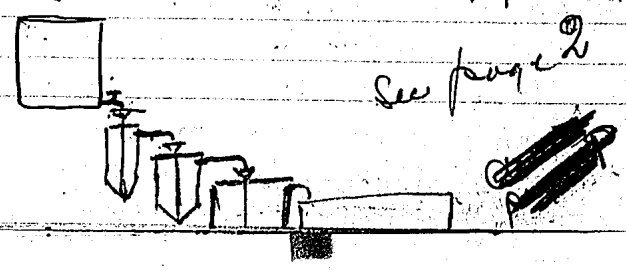
L. Sple #1 Contin.

Weight 1000 gms.
Nothing gained or lost
Passed thru 90 mesh
Oversize 90.754
Undersize 909.746
1000.000

1/10 or 99.98 gms, mixed with about 200 ccs H_2O, allowed to settle for a few moments & finely suspend material taken found to disintegrate electrolytic apparatus — Did not work very well — clogged bottom of each classifier.
(Washed all of above into a beaker to be treated later)

Took 1/10 = 99.98 gm and passed thru 200 mesh screen
Over
Under { Did not work well did not finish }

Passed undersize thru classifier
Exp #1 Used disintegrator apparatus



See page 2

Sple #1 Contin from page 18

Accidentally the large Sple was misplaced, so thought to would try to classify the whole out.

Stirred in large Cassarole, allowed to settle a few moments and poured to large beaker where it was allowed to settle even no long, when it was poured to large settling vessel.

Would not settle properly was forced to filter it. ~~as it~~ passed thru' filter.

Although large quantity of water was used separation was not clean, as I found a fraction, larger than 0.0015" dia.

Some of observations lost
Wt. of Fines (dry) 280 gm = 27.18 %
Water used = 16500 cc
= one ft per Ton fines
= " " per " Rock
Specs put away for future reference
It was simply to compare with Ignition
Contin on page 19

Mch. 15/20

Analyses of Spodumenes from Norwich, Smith and Brush.

SiO_2	64.04
Al_2O_3	27.84
Fe_2O_3	0.64
CaO	0.34
Li_2O	5.20
Na_2O	0.66
K_2O	0.16
H_2O	0.50
Total	99.38

Dana

Dana Analyses of Orthoclase from Bauberg, Cx. Smith & Brush

SiO_2	63.88
Al_2O_3	18.97
MgO	0.20
CaO	0.70
Na_2O	3.78
K_2O	11.19
Loss on Ignition	0.40
Total	99.12

Dana

Mch. 15/20

Analyses of Amblygonite from Paris Me. by Penfield

P_2O_5	48.34
Al_2O_3	33.65
Li_2O	9.84
Na_2O	0.34
H_2O	4.49
F	4.82
K_2O	0.03
Total	101.89
	2.03 deduct for O
	99.86

Dana Analysis of Lepidolite

	Rummelsberg	Cornwall
SiO_2	51.70	57.40
Al_2O_3	26.76	26.80
Mn_2O_3	1.29	Mn_3O_4 1.50
MgO	0.24	
Na_2O	6.15	
Li_2O	1.27	4.85
K_2O	10.29	9.14
F	7.12	4.18
CaO	0.40	
P_2O_5	0.16	
	100.38	98.87

Mch. 15/20

Analysis Glauconite

N-Jersey Hand

SiO_2	50.70
Al_2O_3	8.03
FeO	22.50
MgO	2.16
CaO	1.11
Mn_2O	0.75
K_2O	5.80
H_2O	8.95
Total	100.00

Contin from page M

There was an intermediate product which showed considerable fines

Tot. of Intermediate 320 gm

Took 1/10 = 64.0 gm

and separated fines

by elutriation, current 12950 cc in 45 min

tot of fines 27.848 gm = 13.5 %

~~" " Coarse~~

288 cc per min

~~Tot. of fines gm~~

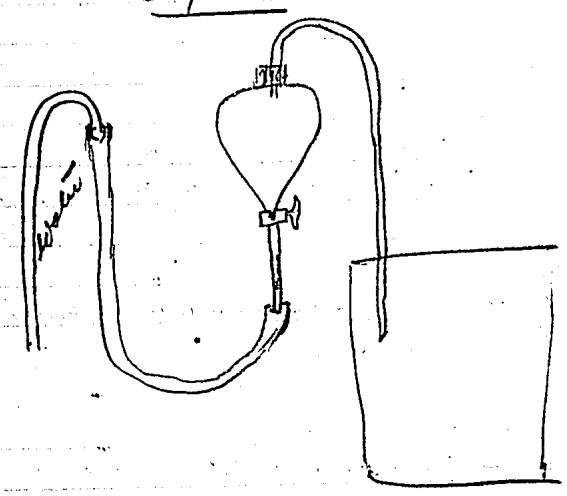
Note all of these #1 precipitate filed away. (See page U)

Contin from page L

Spec #2 Exp #1 (Contin.)

This app. did not work at all. Fines Dem terminated with Dress from app. and thrown away. Coarse was saved by putting it one side.

Exp #2



Took 92.98 gr

Water Current 7700 cc in 2 hrs.

= 64. cc/min.

Fines :- 22.6 gm = 22.6 %

Contin on next page

R

Exp. #2 (Contin.)

This fines high-grade but yields very poor.

Tried ~~[illegible]~~ amount
— cc H_2O in 30 min.
in 3 different jars
Largest particle just about 0.005"

Jar 1	4650	cc H_2O	10.667 grms. Mat'l
" 2	3750	" "	5.313 " "
" 3	4550	" "	1.604 " "
=	12950	= 461 cc/min	

Jar 1	35.90	% of H_2O & 60.6	% Mat'l
" 2	28.96	" " " & 30.21	% "
" 3	35.14	" " " & 9.13	% "

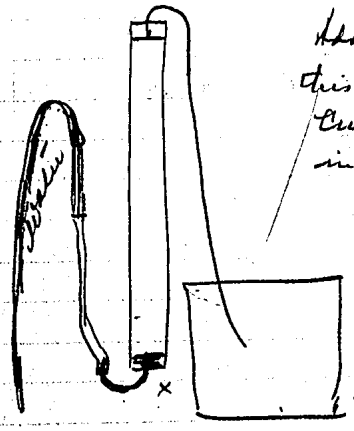
Water used compared with % fines recovered

1 7700	cc =	37.29 % =	22.6	56.94 %
2 4650	cc =	22.52 " =	10.667	26.54 %
3 3750	cc =	18.16 " =	5.313	13.22 %
4 4550	cc =	22.03 " =	1.604	4.00 %
Total 20650			40.184	

Fines Recovery 40.18 %.
Oversize still contained considerable fines — but fines free of oversize

S

Exp # 3



Added 92.98 gr to this tube.
Current 3750 cc in 1/2 hr.

Tot of Fines
= 58.607 gr
= 63.03 %

This viewed work OK with bottom like

This did not work better because water channelled thro' mixture.
— Might work better if x came to a point.

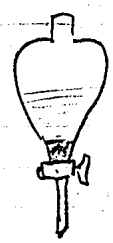
Notices in this that largest oversize might pass from operation at the very outset by dropping against a comparatively rapid current & bottom of a separate app.

Some of the oversize lost.

Rest put to one side

Exp. #4

Took 9.94 gm and washed into separating funnel, filled 1/2 full of water — shook up and allowed to settle a few minutes — Open cock and let coarse run out.



Separation was not clean.
Fines stuck to sides etc.

Discontinued & did not weigh products.

Recovered fines for further treatment

Sample #1 Continued from page P

After filtering, noticed that considerable fog from colloidal material still in suspension

250 cc filtrate =

16.519
16.350
.169

= 0.676 gm per L.
0.0422 # per imp.

After recovery of the 27.848 gm fines on page P — tried a further current 11000 cc in 28 min. 440 cc/m to see how much more could be recovered

8.881 gms. = 4.31 %

Result

	Gds	H_2O %	Mat'l Gms	%
1				
2	~~9.94~~		~~27.848~~	
3	~~11000~~		~~8.881~~	

(Total fines produced = 45.02 %)

4/1/20

Exp #5

Took the remainder of Sper & v.
= 547. grms

Treated by various methods
(See following pages).

obtained 170 grm Coarse
= 377 " Fine
by diff.

or 68.9 % Recovery

Note Some of fine lost in manipulation

Approximately all under 0.0015"

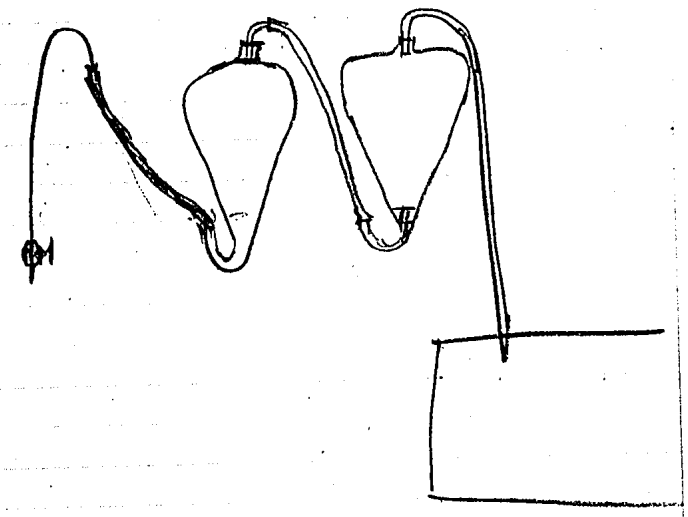
~~Receptacle~~ Crock for reservoir
14.25" dia × 19.75" deep = 3150. cu in

every inch = 159.5 cu in = 2614 cc

14 1/2" in 22 min
= 1163 cc in 22 min
= 56.4 cc per min.

opening for discharge tube
1/4" inside dia.

used this app. for Exp 5



This tandem arrangement used an excess of water pressure changing without warning.

Total wt. of material for regrinding 287 gm

Note This is all from 1000 grm Sper & v

Both fines & coarse were lost in diff ways in manipulating.

Exp # 6

Took 282 grams Overseize
(Split 2)
In Ball-Mill 2 hours.

In	282	
Out	282	
	000	Gained or lost

282 gms powder = 243 cc
or " cu. ft.

Powder	243	cc
+ H_2O	200	cc
= Total Vol	287	cc

= Sp. Gr.

Used Tandem Cone Classifier
Rather rapid overflow
5.75 " in 56 min
= 150.31 cc in 56 "
= 268.4 cc per min.

Note Of course the cap. for Mill is too small.

Continue.

Overseize

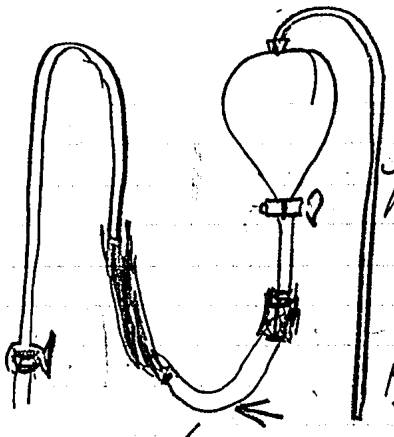
In	282	grams
Overseize	19	"
Fines	263	"
=	93.26	% Recovery

Although under Microscope all of this fine metal seems to be within the limits of 0.0015 it might be good policy to be satisfied with a smaller yield of finer concentrate.

B

A

Did not like the idea of having to agitate so much material in order to make separation of fine and coarse so tried following



Noticed that oversize had tendency to accumulate in place marked by arrow

Glass elbow

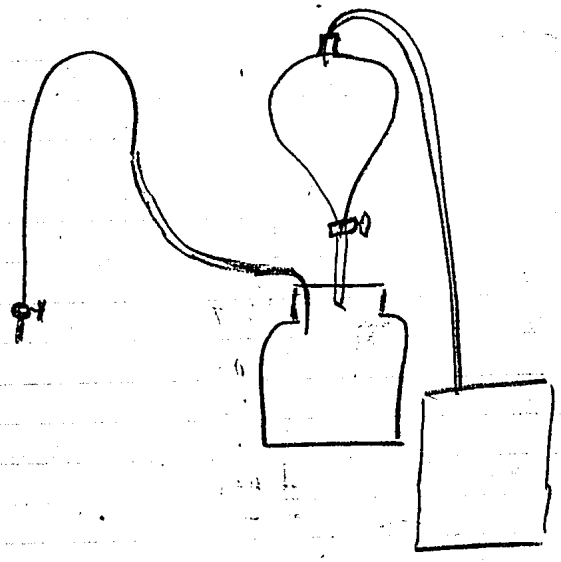
Found that opening in elbow was too small.

N.G.

C

B

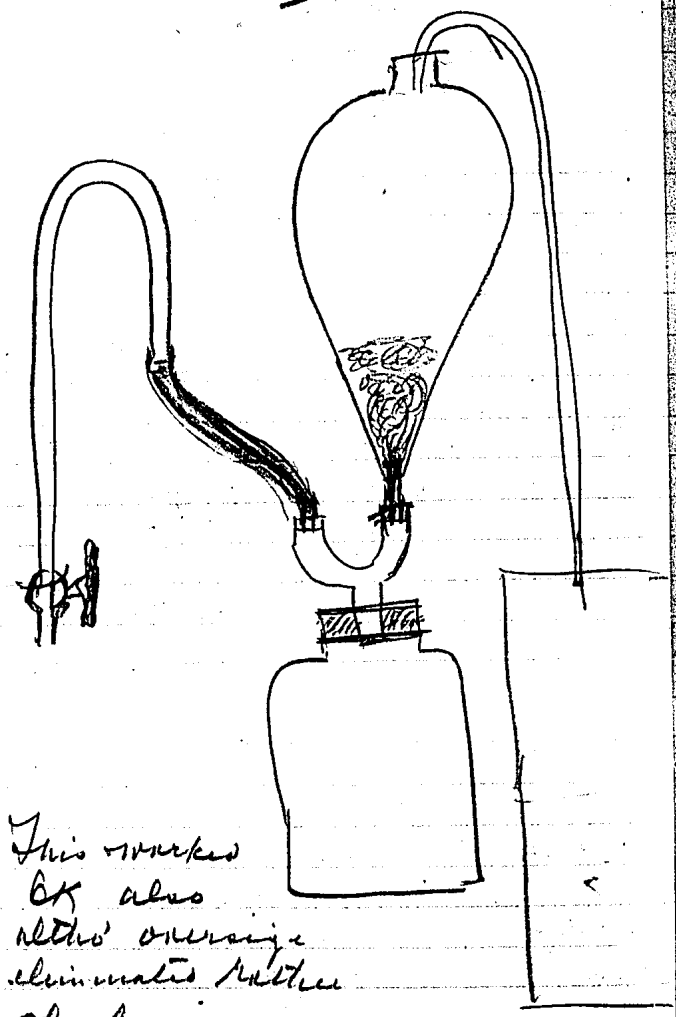
Tried following :—



This showed that if given a channel that the oversize would naturally sink against the counter-current and settle in bottle, but was too slow on account of friction.

D1

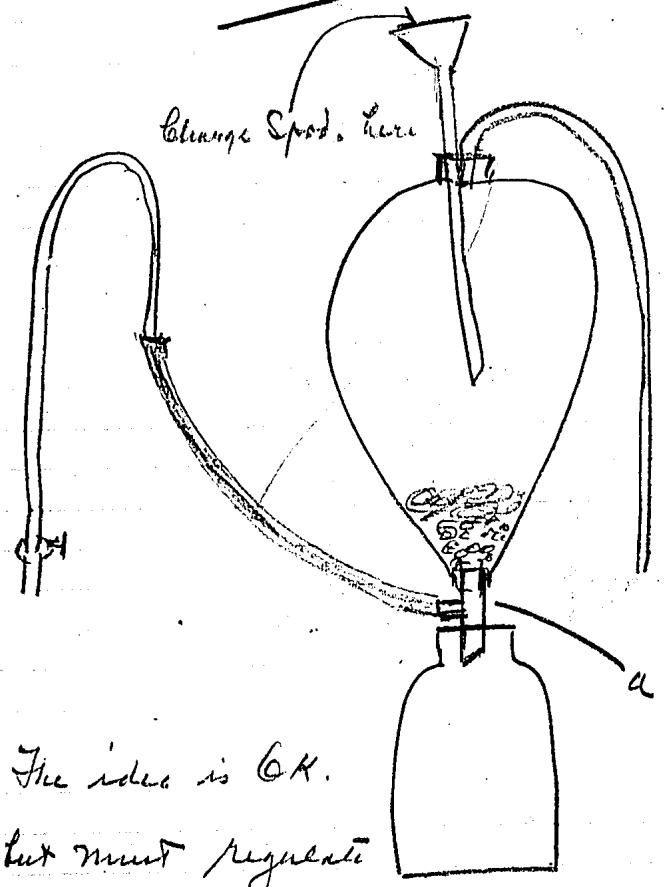
a



This worked OK also altho' overcharge eliminates rather slowly

E1

b



The idea is OK.

but must regulate the flow of water and have opening at "a" proper size.

F1

6

7.

G1

A,

Exp #7

Noticed in looking over Mr. Edison's Notes that Amblygonite seems to break up readily with acids and metallic salts that hydrolyze readily — Looking over analysis of average Amblygonite found it to contain about 4% Fluorine

Suspect that it aids decomposition by continuous formation of HF — H_2SiF_6 in presence of strong mineral acids, which breaks up the silicates, liberating the bases which immediately combining with the acid, and Fluorine compound made avail for further action.

If this is so, by taking a mixture of Amblygonite and Spodumene and adding (say SO_3 H_2SO_4) to combining with bases stones in time extract all of the lithia, Potash, Alumina etc. and leave the SiO_2 behind.

9,

Exp #8

Took equal proportions Spod. and Amblygonite (finely divided) in Erlenmeyer added excess dilute H_2SO_4.

Heated on hot plate for 12 hrs.

Sol. gave decided reaction for Li — K and Al_2O_3

Mixing with Orthoclase lower say, gave heavier K reaction

Looks as though, with time we might appear complete extraction.

Reported to Mr. Edison, that he does not want this line followed, at present at this time.

91

Milk of Lime

Burnt Lime from Drum in Store Room

Slaked and made up 2 litres

Took 100 cc to 1 litre

1/10 = 10 cc =

12.8.6 – 126.1 cc HCl

= I CaO :– 163.06 gm per l.

II " :– 159.89 " " "

= Ave " :– 161.48 " " "

1/10 = 10 cc = 2.0 – 2.0 cc HCl

= I $CaCO_3$ 2.53

= II " 2.53

= Aver " 2.53

Calculate only on actual $Ca(OH)_2$

10 gm Specimen (say 65.00 % CaO)

= 11.93 gm. $Ca(OH)_2$ actual

or 73.9 cc of above

Note Use 75.0 cc for 10 gm Sp.

Stand. 1/2 HCl Sol.

Sp. Gr. HCl = 19.73 @ Room temp

= Sp. Gr. 1.15 = 29.65 % HCl

Take 150 cc for 2 litre

Standardization :–

1) 1.0043 gm Na_2CO_3 = 41.9 cc HCl

2) 1.0069 " " = 42.02 " "

3) 1.0050 " " = 41.95 " "

1) 1 cc = 0.02397 gm. Na_2CO_3

2) 1 cc = 0.02396 " "

3) 1 cc = 0.02396 " "

aver. 1 cc = 0.02396 " Na_2CO_3

1 cc = 0.01653 " HCl

1 cc = 0.01268 " CaO

1 cc = 0.02263 " $CaCO_3$

1 cc = 0.01086 " LiOH

(0.9042 % of 1/2 Strength)

Li

Standard LiOH Sol.
for Comparisons

Lithium Hydrate Crystals
28.4607
27.5417
.9190 wt. of dry LiOH
= 48.3 cc M/2 HCl
= LiOH 57.09 %

15.2290
14.7380 (Clark as Chloride
.4910 LiOH was

K_2O: Trace

15.235
14.7380
.495 LiCl
LiOH 56.95 %

5/7

Aver. 57.02 % LiOH
= 35.57 % Li_2O

10. gm. 6 cc = 0.4 gm. Li_2O.
250 cc = 0.040 gm Li_2O —
Took 9.00 gm. (0.8996 gm. ast)
and dilute to 2000 cc
1/10 = 0.900 to 2000 cc = 0.0400 Li_2O per 250 cc

Entire analysis is made total as Li_2O as 4.0 % = LiOH 6.41 % { 0.00016 gm Li_2O per

See page 21

Memo

Potash Aluminate $K_2Al_2O_4$
Soda " $Na_2Al_2O_4$

Silico-Aluminate Soda
$(Na_2O)_2(SiO_2)_2Al_2O_3$
$(K_2O)_2(SiO_2)_2Al_2O_3$
$CaO(SiO_2)_2Al_2O_3$

Calculating to
Calcium Mono-Silicate
1 % element in Rock =

CaO	=	1.08 %	SiO_2
MgO	=	1.40 %	"
Fe_2O_3	=	0.38 %	"
Al_2O_3	=	0.59 %	"
K_2O	=	0.64 %	"
Na_2O	=	0.97 %	"
Li_2O	=	2.02 %	"
SiO_2	=	0.93 %	CaO

The SiO_2 required for CaO, MgO & Fe_2O_3 is deducted from total SiO_2 & remaining SiO_2 multiplied by 0.93

No 1

Memo.

$2\,CaO + SiO_2 = Ca_2SiO_4$
$2\,MgO + SiO_2 = Mg_2SiO_4$
$Fe_2O_3 + SiO_2 = Fe_2SiO_5$
$Al_2O_3 + SiO_2 = Al_2SiO_5$
$K_2O + SiO_2 = K_2SiO_3$
$Na_2O + SiO_2 = Na_2SiO_3$
$Li_2O + SiO_2 = Li_2SiO_3$

1% Element in Rock combined with SiO_2 as follows

CaO	=	0.54	SiO_2
MgO	=	0.75	"
Fe_2O_3	=	0.38	"
Al_2O_3	=	0.59	"
K_2O	=	0.64	"
Na_2O	=	0.99	"
Li_2O	=	2.02	"
SiO_2	=	1.65	CaO

Di-Basic Silicates

Analyses of Specimens (Edwards)

Note: This is Sample # 6, and had been calcined and powdered in Gold mortar.

1.0 gram = 0.627 – 0.6208 gm SiO_2
SiO_2 = 62.0 – 62.08 were 62.24 %

1.0 g 0.28 – 0.28 gm $Fe_2O_3 + Al_2O_3$

1.0 g 2.3 – 2.3 as K_2CaMgO_4
= Fe_2O_3 = 1.53 % 1.53
= Al_2O_3 = 26.37 – 26.57 – 26.47

1.0 gm 1.4 – 1.4 as $KNaMgO_4$
= CaO 0.39 %

2.0 gm 17.7400
17.7265
.0135 $Na_2P_2O_7$
= MgO 0.25

5/6

60.1830
59.9030
.2800

3[illegible].3077
3[illegible].0770
.2307 .2307 % – 21.00%

LiCl + NaCl + KCl

3[illegible].5[illegible]
3[illegible].0770
.4[illegible]5

60.1[illegible]
59.908
.2[illegible]

17.7195
17.7105
.0090
KCl 0.09 %

20 gm used
Li K Na Cl = LiCl = Li_2O = 3.89
Mn = trace K_2O = 0.09
P = " Na_2O = 5.59

Total 100.6 %

1 Ton 2000# = 1274.7 # Li Cl

P.

Exp #9

5.0 grms Spod.
40.0 cc Milk of Lime

Erlenmeyer Flask & Funnel.

Hot-plate for 24 hrs
Stirring occasionally

Filter - (filtered readily)

Filtrate to 100 cc
gave just a flash of Lithia

Ppt showed HCl sol SiO_2 but
no Al_2O_3

N.9.

Exp #10

5.0 grms Spod.
40. cc Milk Lime

Erlenmeyer Flask & Funnel

Hot plate for 2 days (48 hrs)
occasionally stirring

Showed greater tendency to pack
than Exp #9

Filtrate to 100 cc
gave more decided
Lithia reaction than #9

HCl sol. SiO_2 much heavier
than #9, and away
out of proportion to the Lithia in sol.
trace of HCl sol Al_2O_3
HCl sol Lithia very bright
as compared with water sol. Li.
Not enough to bother measuring
but shows decomposition takes place.

N.9.

R.

Notes

Exp #9 010 shows that it may take quite some time to break up in this manner.

Must be kept in motion.

Covered if possible, for CO_2 will destroy activity of $Ca(OH)_2$

Heat is quite essential.

Exp #11

5.

Took 2 portions 5 grams each + 40 cc Milk of Lime in Erlenmeyer as with Francis. On hot plate 48 hrs.

a	b
5. gms Spod.	5. gms Spod.
40 cc Milk Lime	40 cc Milk Lime
	2 cc 7½ NaOH

Added a little water to keep up vol.

(a) About same as #10
Rather granular

(b) Darker looking than (a)
More like putty.

Although in (b) more thorough decomposition was shown, since little reaction was no thorough, but the sol. $Li - Al_2O_3$ & Li_2O, much thorough than (a)

Apparently SiO_2 alone is taken up first by $Ca(OH)_2$.

N.G.

Exp #12

Exp #11 showed small addition of NaOH acted as an accelerator.

Took
Spod. 5 grams
NaOH more than enough to combine with all SiO_2.

Put in Erlenmeyer flask—funnel on hot-plate 24 hrs.
Shake occasionally

Material seemed to swell, and lost its granular appearance.
Filters readily.
Sol. showed SiO_2 in sol.
Did not give a good Li line.

Adding Milk of Lime extracts the SiO_2 from Soda sol. regenerating NaOH for further use.

[redacted]

Question.

I think this is worth further investigation as NaOH is no doubt much more active in decomposing refractory Silicates.

After filtration, the SiO_2 could be removed and NaOH regenerated in another vessel by means of $Ca(OH)_2$.

This has the advantage also of not packing so much, as it allows of a thinner slurry.

Look into this further later on, as it could be applied to Feldspars also.

Strange results, looks as though only the SiO_2 goes into sol. Concentrating the Li, Al, K etc in the insol.

Acidifying with HCl, gelatinous the residue same as slags, showing high yields of Li, Al_2O_3 - SiO_2 and a plant [redacted]. ENCOURAGING.

1

Exp #13

5.0 gms Spod.
50.0 cc Milk of Lime
5 drops 1/2 NaOH Sol

Erlenmeyer Flask – Funnel Cover
Hot – plate, 36 hrs
accompanied shaking.

Color darkened.
Filtered easily.
Filtrate to 250 cc
Took 63 cc to 250 cc
Lithia flask 30 seconds
Mr Edison Standard
40 mg to 250 cc 40 "

LiOH extracted 40 mg LiOH × .75
= 30 mg LiOH
= 18/10 " Li_2O
As 5.0 gm = 0.206 gm Li_2O
yield = 3.40% Li_2O

See note on opp. page

Note

Discontinue this line of investigation until assay of Spods. is completed and Standard LiOH has been checked.

Also study Spectroscopic determinations, even on large numbers to obtain average length of flames, and other standard conditions.

Notes on #9 – #10 – #11 #12 & #13

Examination under Microscope and comparing before and after treatment with $Ca(OH)_2$ etc. showed that whereas, before the particles were smooth on the surface that after treatment, the surface was very irregular and pitted in appearance.

Exp. #14

Took solid piece CaO
= 11.5 gm + 11.5 gm Spod.

Ground it powder in Mortar & pestle
Mix thoroughly with Spod. —
placed in porcelain Crucible
(small) — Moisten with H_2O —
much heat generated, and mass
solidified like Mortar. ~~[illegible]~~
allowed it set for ½ day then on
hot plate for ½ day — Quite
Solid.

~~Stood~~ in Lab. for 3 days altogether
Could not remove it very
easily with knife. ——

Had to grind. moist in Mortar &
pestle broke up readily.

1 Boiled first of all with water filt
2 Added Dilute HCl to residue until
all free lime was neutralized
as shown by Phenol phthalein filt
3 Added xs HCl to residue to
see if any further extraction
was possible.
4 Dried residue & took 1/10
wt = 8.9 grms 1/10 = 0.89.

11.5 gm
8.9
2.6 gm. gm loss in treatment
or 22.74 %

Fractions

1/600 cc to	7200 cc	with H_2O	=	0.[illegible]
1/250 cc "	1500 "	" "	=	0.[illegible]
1/250 cc "	3400 "	" "	=	0.[illegible]
0.89 gm =	5000 "	" "	=	0.[illegible]

Assayed Insol. residue after
treatment with xs of HCl and
found it to contain
SiO_2 :- 51.63 %
as compared to

31

Contin. from page L1

1st Trial 11.3 + 0.9 = 12.2 cc LiOH Sol
{ 0.0400 gm Li_2O per 250 cc
= 0.00016 g Li_2O per cc
Showed Li line at 500 cc dilution
or 0.00016 × 12.2 = 0.00195 ÷ 500
= 0.000003904 6 grms per cc

2nd Trial
10.0 cc Lithia (.0400 Li_2O per 250 cc)
Showed Lithia after dilution to 500 cc
= 0.0000032 grms per cc

N.G.

Check on
Stand LiOH Sol. on Chalf.
marked :-
0.04 grms per 250 cc
or 0.00016 grms LiOH per 1 cc
20 cc = 0.0032 grms. LiOH

Diluted with Water to 570 cc
just showed Li line
= 0.000005614 grms LiOH
= 0.0000035 02 " Li_2O
per 1 cc

Show according to
preceeding that
this is marked approximately
9.4 % too high

Check these figures again
on following page
N.G.

E.E.D. Standard

Mark 0.0400 gram Li_2O per 250 cc

30.0 cc + 1000 cc H_2O.

M.G.

Old Standard used in previous work

40.0 cc + 570 cc H_2O.

M.G.

Final Standard E&S

25.0 cc = (0.00016 gram act. Li_2O per cc)
or 0.04 gram per 250 cc
slightly acid with HCl :—
25.0 cc + 550 cc H_2O = 575 cc
Li line just dis appeared.

.00016 × 25 = 0.004 gram Li_2O
0.004 gram Li_2O ÷ 575 =

1 cc = 0.0000069 57 gram Li_2O
× 575 =
0.040 gram Li_2O
0.0641 " LiOH
per 250 cc

5/7

Actual Standard

(Li_2O + 2LiOH × 1.603)

Final Check vs. Old Standard

25.0 cc + 465 cc H_2O = 490
Li line just disappeared.

Using my Standard then
Act. =
1 cc = 0.00000593 gram Li_2O

× 490 = 0.02906 gram Li_2O
= 0.04658 " LiOH

or 72.65% of value
of my Standard

5/7

Calculations

Exp #15

10 gram Spod.

75 cc Milk of Lime = 12.1 g $Ca(OH)_2$

5 P.M. 5/7 to 10 A.M. 7/10 = 65 hrs

Placed in Erlenmeyer flask with funnel on hot plate — Cooked and began to crack — Stiff mixture that stuck to sides of flask also on stem of funnel when it had spattered. — Did not disintegrate upon addition of water. — Material had gray color except at bottom & centre where it had a ruddy color.

Gave impression of having decomposed the rock, but no doubt action had stopped through lack of agitation & the accretion of rock.

Broke up readily with water in mortar & pestle. Filtered — Filtered

1) Water extract & washings = 300 cc
dilute to 4550 cc then took 200 cc and dilute to 420 cc before lime disappeared.
400 : 420 :: 4850 : x = 1085 cc
= 0.0709 gm. Li_2O

2) Dissolved the residue in xs HCl & Filtered — Filtered

Exp #15 Continued.

poorly — ~~Filtrate~~ Filtrate and Washings 300 cc
1/3 or 100 cc took 1900 cc water
= Total 2000 cc × 3 = 6000 cc H₂O
= 0.0418 gms Li_2O
Acid sol. showed considerable Al_2O_3 in sol.
(Threw reaction out)

Result in	Li_2O	
10 grams =	0.389 gms	100
water	0.0709 "	18.25
HCl	0.0418 "	10.70
Total.	0.2763 " Lost	71.01

Acid sol. test Must simply to see if most was decomposed and to determine ~~anything~~ the time held, by dissolving the lime.

Exp #16

10 cc Mg(NO₃)₂ Sol. — 0.2056 MgO
10 gms Spodumene
75 cc. Milk Lime
Same Conditions as #15
Same time " 15

Cooled Same as #15 — also showed Residue Spot in Crucible at bottom — Did not disintegrate by boiling with water. — Broke up Ox. in Mortar & pestle. Filtered — Followed Carefully

Washings × Washings = 500 cc
Took 1/5 = 100 cc & added 2400 cc water
= 3000 cc Total × 5 = 15000 cc
= 0.1044 gms Li_2O

Check by separating & weighing as LiCl, the Li in the other 4/5

32.1477
32.0977
.0625 LiCl = 0.08125 LiCl gms in all.
= 0.0786 ✓ gms Li_2O in 10 gms

Note

Looks as though a quantitative Method must be worked out for this work

Exp #16 Contin.

2) HCl sol. = 250 cc sol.
Dilute to 1000 cc Took 25 cc
and added 275 cc H_2O to reach
initial point
25 + 275 = 300 cc Total
25 cc : 300 :: 1000 x
x = 12000 cc
= 0.0835 gm Li_2O

Total		0.389	100. %
N. Sol	0.1054		26.83 %
HCl Sol	0.0835	0.1879	21.46 %
Insol.		0.2011	51.71 %

Acid Sol. showed considerable
Al_2O_3 in sol.

Exp #17

10 gms. Spodumene
+ 13 " Rock (99% Dolly)
+ 75 cc H_2O

Treated identically the same
as Exp. 15 & 16 and for
same length of time, — &
the exposition of time, did
not seem to have lost water,
nor did it stick, whereas 15
& 16 caked, and stuck to sides,
mixture pale brown in color,
filtered easily. Solids appeared
to have been little acted upon.

Filtered —
Ext. & Washings 315 cc
Took 25 cc and diluted to 500 cc
= 525 cc Total
(Note Cola marked Li line toward end)
25 cc : 525 :: 315 : x
= 6615 cc
= 0.046 gm Li_2O

2) HCl in excess — Filter
Filtrate & Washings = 300 cc
Made to 1000 cc took 50 and added
1200 cc = 1250 cc Total cc
= 0.1670 = Total in 25000 gm Li_2O

Exp #17 Caustic.

Total		0.389	100.00
W-Sol	0.046		11.8%
HCl Sol.	0.167	0.213	42.93
Insol.		0.176	45.25

Adding $Ca(OH)_2$ to the W. sol. precipitates the SiO_2 very readily — Ppt filtered easily on coarse filter cloth — & did not contain very much free $Ca(OH)_2$ or NaOH.

If this proved efficient, the mixture of LiOH & NaOH caused the regenerate to be used over again until LiOH contents became so high that it caused the xtallizes out on separation by other means

5/15/20

Preparation of 4 Grades Fine Spodumen

Understood Mr. Edison obtained good results from flotated material up to 0.0015" dia. ——— Batch for experimental purposes approximate this size, but seems to be the trig. Could not find any of Spod. that Mr Edison worked with, but a sp. of flotated Amblygonite that Mr. E had worked with approximated my material in size i.e. maximum = 0.0015" dia.

Took 200 grms. flotated Spod. and classified as follows

1	Stage 1/5	Fraction	4.87	=	194	Min
2	" 4/0	"	5.50	=	70	"
3	" 8/5	"	10.00	=	75	"
4	" Residue	"	—			

(Outlet orifice = 1/8" dia.)

1	4180	cc Water	= flow	40.0	cc per Min
2	8580	" "	=	172.6	" "
3	31600	" "	=	421.3	" "
4	—				

Results.

	Tot	%	Size	
1)	35.3 gm = 17.65 =	100-60	4/10000 –	cc/1000000
2)	26.5 " 13.25 =	60-20	6/1000000 –	133/1000000
3)	65.5 " 32.75 =	30-10	133/1000000 –	4/10000
4)	72.7 " 36.35 =	10-3	4/10000 –	133/100000

Particles across space 0.004" on Dia. Record

Note In each class there was also small proportion of very fine material.

Calculated on original Starting from 1/4" Size (1000 grms.)

1)	172.64	grms =	17.26	%
2).	91.34	" =	9.13	"
3)	225.77	" =	22.57	"
4)	250.52	" =	25.05	"

and after 3 hrs. grinding

Calculated on Double grinding 5 hrs.

1)	136.36	grms =	13.63	%
2)	102.79	" =	10.23	%
3)	257.83	" =	25.78	%
4)	280.62	" =	28.06	%
Total	772.00		77.20	%

Average of 263 gm from 282
and " 377 " " 547
= 77.20 % yield

5/20/20

Calculation of CaO necessary for decomposing Spodumene
Ortho-Ionic Silicate
(For 100 gmm)

						SiO_2
Li_2O	3.89	×	2.02	SiO_2	=	7.86
K_2O	0.09	×	0.64	"		0.06
Na_2O	5.59	×	0.97	"		5.42
CaO	0.39	×	0.54	"		0.21
MgO	0.25	×	0.75	"		0.19
Fe_2O_3	1.83	×	0.38	"		0.70
Al_2O_3	26.47	×	0.59	"		15.62
SiO_2	62.14	×	1.65	CaO		30.06
					SiO_2 in Ambin	30.06

6.15 − 30.06 = 37.08 to be taken care of by CaO

37.08 × 1.65 = 57.93 gmm CaO to be added, or 5.79 for 10 g

Milk of Lime = 161.48 gmm per liter
or 37.7 cc
Use 35.0 cc

Exp #18

10 gmm. Spod. Size #1
35 cc Milk Lime
+ 50 cc H_2O

Digest at from 65°C – 80°C for 5 hrs. — Constant Stirring.
— At end of 5 hrs. heated up to 100° finishes.
Transferred to beaker & heat.
— Settles quickly, liquor clear although $Ca(OH)_2$ in excess. —
Filtered readily on ordinary paper.
— Filtrate about .75 liter.
Water sol. & Washing is made all except to dry weight equal.

59.9605
59.9055
.0550 Li_2CO_3

Li_2O 0.0149 %
" 0.15 %

Take remainder shook up in 500 cc flask 1/10 = 44 cc 14 cc

~~Dist~~

Ex 18 Carlin

Dissolving in HCl liberates considerable Al_2O_3 and Lithia.

Insol. Residue = 8.80 gm

1.0 = 32.1500
32.0773
.0727 Li_2O.
= Li_2O 1.43 %

~~Dilute to 1200 cc = Li_2O = 1.50 %~~

→ Dilute to 840 × 2 = 1680 cc
= 0.00445 gm Li_2O
= 0.45 % in Insol. Li_2O

Original =
1.0 gm – ~~32.2475~~
~~32.0770~~
~~.138~~
~~Li_2O 3.75 %~~
32.199
32.077
.122
Li_2O 3.32 %

Exp #19
10 gmm Spod Size 2
35 cc Milk of Lime
10 cc H_2O

Digest at 60°C - 80°C for 5 hrs. — Constant Stirring — At end of 5 hrs. had a pretty book, which did not settle very readily — Transferred to beaker and treated — Settles and filters easily — Washed easily — $Ca(OH)_2$ in sol.

Water sec & nitric evap. & contained
59.9420
59.9055
.0365 Li_2O.
= Li_2O :- 0.0099 gm
= Li_2O :- 0.80 %

Dissolving in HCl liberates considerable Li & Al_2O_3.
Residue under microscope highly flitted an impure

→ Dilute to 400 cc Li line 5 sec.
= Li_2O :- 0.05 % gr. Sol.

Exp 19 Caution

Residue from HCl treatment
= 9.15 gm

1/10 - 59.0470
59.9035
.0435

Li_2O 1.18 %

~~Dilute to 550 cc with H_2O =~~
~~= Li_2O :- 0.64 %~~

Dilute to 1000 cc then 1/2 to 470
= 1880 cc Take
= 0.00498 gm Li_2O
= 0.50 % Li_2O in Fusel.

Original 1.0 g = ~~60.067~~
~~59.9030~~
~~.164~~ ~~Li_2O 0.~~

60.067
59.903
0.164 Li_2SO_4
= Li_2O :- 4.46 %

~~Li_2O 4.13~~

Exp 20

10 grams Sprod. Sig. #3
35 cc Milk of Lime
10 cc NaOH Sol = gm NaOH
35 cc H_2O

Digest 68°C - 80°C for 5 hrs. with constant stirring — Look frothy & did not settle readily — Transferred to beaker & boiled — Settled — filtered & washed readily. — $Ca(OH)_2$ in sol.

Note Stand warmed for about a week, when water evap. off and sediment solidified — did not break up on boiling with fresh water, — Had to grind in mortar when it broke up readily

Evap. W. Sol & Weighing to
59.9358
59.9035
.0323 Li_2SO_4
= Li_2SO_4 :- 0.00878
Li_2O :- 0.0878 % estimate

Extraction of water insol with HCl shows large amt of HCl sol. Lithia & Al_2O_3

Cont'd 20

Caution

Residue from Hcl Treatment
= 9.40 g

1/10 = 59.9400
59.9005
.0395 Li_2SO_4

+ 250 cc H_2O & Took 95 cc & added to 200 cc H_2O = 295 cc Total
= 0.000246 gm Li_2O
= 0.043 % Li_2O on original

In Beaker + 250 cc H_2O, 85 cc added to 1000 cc
= 0.00751 gms Li_2O
= 1.94 % Li_2O
= Total Li_2O left in residue 1.94 %

Orig. 1.0 gm. 60.0940
59.9005
0.0935 Li_2SO_4
= Li_2O 2.54 %

Check
Made to 250 cc — 90 cc of this added to 3300 cc H_2O = 2.49 % Li_2O

Standard

Took 25 cc Stand Li_2O Sol.
(40 mg Li_2O per 250 cc)
= 0.004 Li_2O and diluted to point where Li line showed 5 seconds

Point reached was 320 cc

320) .0040000 (.0000125
320
800
640
1600

1 cc = 0.0000125 gm Li_2O

Do not use this Stand.
as adding sol. to measure quantity of water until Li line shows, has been adopted.

Batch: 21

Size #1 :- $\frac{17.65}{10}$ gm * 3 = 5.295 g
" 2 :- $\frac{13.25}{10}$ gm * 3 = 3.975
Total 19.270 gm

+ 75 cc Milk of Lime
+ 10 cc H_2O

To 70°C over, for 24 hrs.
with stirring

Contin.

Butter 2v

Sign 11-17.65 × 2 = 2.942 gm.
" 2 - 13 3/25 × 2 2.209 "
" 3 - 32 3/25 × 2 5.458 "
3 Taken 10.609 "
+ 80 oz Milk of Lime
+ 10 oz Water

Standard
Use this instead of all others

25 cc Stand Li_2O Sol = 0.004 gm Li_2O

dilute to 130 cc with distilled H_2O
1 cc = 0.00000093 gm Li_2O
showed line for 5 seconds

Took 400 cc of above to add to 1000 cc water to quit show Li line.

0.00000093 × 400 ÷ 1400 =
0.000000265 gm Li_2O per cc

8/5/20
Specs of Weathered Spodumene from Mr Edison
taken by Mr Mallory, Sourenzy

1) Semi-hard Spod from Open cut #1 A
2) " " " " " " 1 B
3) Soft, decomposed Spod " " " 1
4) Very Soft, " " " " " 1
5) " " Spod. from bench, South of open cut #1
6) #6 Semi hard Spod 6.C. #1
7) Semi hard Spod. from bench South side open cut #1
8) Rock which lies near the Spod. logs and which may contain lithia

Each one broken to pass 10 mesh
Composite Sple of 5 gms each to 110 mesh
1.0 gm used for assay :-
Fuse to 250 cc Took 50 cc = 1/5 or 0.2 gm
& diluted to 3080 cc
= Li_2O :- 4.08 %